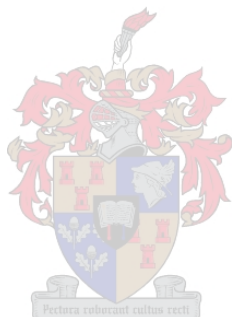


# **Development of value-added dried pomegranate arils and juice powder: Effects of cultivar, harvest maturity and storage duration of whole fruit**

by  
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*Dissertation presented for the degree of Doctor of Philosophy,  
(Agricultural Sciences), Department of Horticultural Science,  
in the Faculty of AgriSciences at Stellenbosch University*

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December 2020

The financial assistance of the National Research Foundation (NRF) towards this research is hereby acknowledged. Opinions expressed and conclusions arrived at are those of the author and are not necessarily to be attributed to the NRF.

## **DECLARATION**

By submitting this thesis electronically, I declare that the work contained therein is my own original work, and that I have not previously in its entirety or in part submitted it for any qualification. That I am the sole author thereof (save to the extent explicitly otherwise stated), that reproduction and publication thereof by Stellenbosch University will not infringe any third party rights.

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## Summary

In South Africa, the increasing rate of pomegranate fruit production, processing and research has witnessed tremendous growth due to consumer interest in its high concentration of bioactive compounds. However, the edible part of the fruit (arils) has a short shelf-life of five to seven days. Agro-processing through drying reduces postharvest losses, improves the shelf-life and increases the storability of the product. Before drying, several pre-treatments, are carried out to preserve the quality attributes of the product, however, little is known about the effects of blanch-assisted drying of pomegranate arils and the prospects of developing high quality pomegranate juice powder (PJP) for formulation and/or fortification to promote product diversification within the agro-processing industry. Therefore, the overall aim of this study was to develop value-added and shelf-stable dried products from pomegranate aril with potential for multiple applications and to provide science-based tools for processing and preservation of the nutritional components. This thesis is divided into seven papers that is organised into four themes. Theme A comprised of the general introduction and comprehensive literature review.

Theme B (Papers 1, 2 and 3) investigated dried arils of three pomegranate cultivars (Acco, Herskawitz and Wonderful) for optimum quality attributes. Paper 1 shows that ‘Wonderful’ had 8.1% and 22.4% higher total soluble solids (TSS) than ‘Herskawitz’ and ‘Acco’, respectively. In Paper 2, dried arils of fruit at commercial and late harvest had significantly ( $p < 0.05$ ) higher TSS than at early harvest. Paper 3 studied the effects of cold storage of whole fruit (cv. Wonderful) harvested at commercial maturity. This paper showed that after 12 weeks of cold storage, arils dried in hot-air had better colour retention based on total colour difference, ( $TCD = 3.02$  vs  $23.6$ ) and retained 46% higher TSS compared with freeze-dried arils.

Theme C provided information on the process optimization of dried arils. In Paper 4, blanched samples of ‘Wonderful’ (7 h) ‘Acco’ (7 h) and ‘Herskawitz’ (8 h), had shorter drying times than unblanched samples (11, 15 and 20 h), respectively. The results from Paper 5 showed that blanching at  $90^{\circ}\text{C}$ , 30s and  $100^{\circ}\text{C}$ , 60s reduced enzyme activity by 76% and 68%, respectively, compared to unblanched samples; this also indicates less browning of blanched arils.

Development of value-added juice powder with multiple applications in the food industry and extended shelf-life (Theme D, Paper 6), showed that juice powder made with maltodextrin appeared 44% redder ( $a^*$ ) than with gum arabic. Similarly, total anthocyanin retention was 54%

higher in maltodextrin than waxy starch. In Paper 7, results showed that at the end of 12 weeks storage, PJP packed in aluminium foil laminated pouches (AFLP) had lower moisture content (6.1%) and water activity (0.54) than other packaging materials, indicating better storability of PJP packed in AFLP.

In conclusion, the quality of dried arils from ‘Wonderful’ at commercial harvest maturity was retained better than other cultivars and harvest maturities. Also, blanching at 90°C, 30s retained the quality of dried arils and was thus recommended as a viable aril pre-treatment condition. Finally, maltodextrin produced PJP had better quality retention compared to gum arabic and waxy starch.

## Opsomming

In Suid-Afrika het die toenemende tempo van die produksie, verwerking en navorsing van granaatvrugte geweldige gegroei as gevolg van die belangstelling van verbruikers in hierdie vrug se hoë konsentrasie van bioaktiewe verbindings. Die eetbare deel van die vrugte (pitte) het egter 'n kort rakleef tyd van vyf tot sewe dae. Agro-verwerking deur middel van droging verminder na-oes verliese, verleng die rakleef tyd, en verbeter die produk se bergbaarheid. Voor die droging van die pitte, is verskeie voorbehandelings uitgevoer om die kwaliteitseienskappe van die produk te behou. Die effek van blansjeer-gesteunde droging van granaatpitte is onbekend, net soos die vooruitsigte om hoë gehalte granaatsappoeier (GPS) te ontwikkel vir formulering- en/of fortifiseringsdoeleindes, om sodoende diversifikasie in die landbouverwerkingsbedryf te bevorder.

Gevolgtrek was die oorwegende doel van hierdie studie om waardetoevoegende en rakstabiele gedroogde produkte van granaatpitte te ontwikkel, wat vir veelvoudige toepassings nuttig kan wees, en om wetenskaplike hulpmiddels te verskaf vir die verwerking en bewaring van die voedingskomponente. Hierdie proefskrif is verdeel in sewe hoofstukke wat vier temas vorm. Tema A bestaan uit die algemene inleiding en omvattende literatuuroorsig.

Tema B (Hoofstukke 1, 2, en 3) ondersoek die gedroogde pitte van drie granaatkultivars (Acco, Herskawitz en Wonderful) vir optimale kwaliteitseienskappe. Hoofstuk 1 wys dat 'Wonderful' onderskeidelik 8,1% en 22,4% meer totaal oplosbare vaste stowwe (TOVS) as 'Herskawitz' en 'Acco' bevat. In Hoofstuk 2 het gedroogde granaatpitte wat op kommersiële en laat oes rypheid gepluk is, beduidend ( $p < 0,05$ ) hoër TOVS as vroeër geoesde granate getoon. Hoofstuk 3 bestudeer die effekte van koel berging op heel vrugte (kv. Wonderful) wat op kommersiële volwassenheid geoes is. Hierdie hoofstuk toon aan dat granaatpitte wat in warm lug gedroog is, na 12 weke van koel berging hul kleur beter behou ten opsigte van totale kleurverskil ( $TKV = 3.02$  teenoor 23.6), en ook oor 46% meer TOVS beskik in vergelyking met gevriesdroogde pitte.

Tema C verskaf inligting oor die prosesoptimalisering van gedroogde granaatpitte. Die droogtye in Hoofstuk 4 van geblansjeerde 'Wonderful' (7 h) 'Acco' (7 h) en 'Herskawitz' (8 h) monsters was onderskeidelik korter as ongeblansjeerde monsters (11, 15 en 20 h). Die resultate van Hoofstuk 5 het gewys dat blansjering by  $90^{\circ}\text{C}$ , 30s en  $100^{\circ}\text{C}$ , 60s ensiemaktiwiteit met

onderskeidelik 76% en 68% verlaag het in vergelyking met ongeblansjeerde monsters; dit dui ook op minder verbruining van geblansjeerde pitte.

Ontwikkeling van waardetoegevoegde sappoeier met verskeie toepassings in die voedselindustrie en 'n verlengde rakleef tyd (Tema D, Hoofstuk 6), dui aan dat sappoeier met maltodekstrien 44% rooier ( $a^*$ ) as dié met Arabiese gom vertoon. Net so was die totale retensie van antosianiene 54% hoër in sappoeier met maltodekstrien as wasagtige stysel. In Hoofstuk 7 het GSP verpak in aluminiumfoelie gelamineerde sakkies (AFLS) aan die einde van 12 weke se opberging 'n laer voginhoud (6.1%) en wateraktiwiteit (0.54) gehad as ander verpakkingsmateriaal, wat beter stoorbaarheid van GSP in AFLS verpakking aandui.

Ten slotte is die kwaliteit van gedroogde granaatpitte van 'Wonderful' op kommersiële oesrypheid beter behou as ander kultivars en oestye. Ook, tydens die blansjering by 90° C, het die gehalte van die gedroogde granaatpitte behoue gebly en word dit dus aanbeveel as 'n lewensvatbare voorbehandeling. Om af te sluit het maltodekstrien-geproduseerde GSP 'n beter gehalte retensie gehad in vergelyking met Arabiese gom en wasagtige stysel.

This dissertation is dedicated to my beloved parents; Mr Adedokun and Mrs Ayodele Adetoro

## LIST OF PUBLISHED MANUSCRIPTS

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1. **Adetoro, A.O.**, Tsige, A.A., Opara, U.L. and Fawole, O.A., 2020. Mathematical modelling of blanch-Assisted drying of pomegranate (*Punica granatum*) arils in a hot-air drier. *Processes*, 8, 611 (IF: 2.753).
2. **Adetoro, A.O.**, Opara, U.L. and Fawole, O.A., 2020. Effect of Carrier Agents on the Physicochemical and Technofunctional Properties and Antioxidant Capacity of Freeze-Dried Pomegranate Juice (*Punica granatum*) Powder. *Foods*, 9, 1388 (IF: 4.092).
3. **Adetoro, A.O.**, Fawole, O.A., Opara, U.L., 2020. Effect of Hot-Air and Freeze-Drying on the Quality Attributes of Dried Pomegranate (*Punica granatum* L.) Arils During Long-Term Cold Storage of Whole Fruit. *Agriculture*, 10, 493 (IF: 2.072).
4. **Adetoro, A.O.**, Fawole, O.A., Opara, U.L., 2017. Effects of pretreatment and drying on the quality attributes of fruit. *Acta Horticulturae*, 1201, 1-6.

## LIST OF CONFERENCE PRESENTATIONS

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5. **Adetoro, A.O.**, Fawole, O.A., Opara, U.L., 2019. Effect of blanching on the physico-mechanical properties of hot-air dried pomegranate arils (cv. Acco). 23rd SAAFoST Biennial International Congress and Exhibition, 1 – 4 September 2019, Johannesburg, South Africa.
6. **Adetoro, A.O.**, Fawole, O.A., Opara, U.L., 2019. Effect of carrier agents on the biochemical activities and rheological properties of freeze-dried pomegranate juice powder (cv. Acco). 2nd All Africa Postharvest Congress and Exhibition, 17 – 20 September 2019, Addis Ababa, Ethiopia.

## SUBMITTED MANUSCRIPT

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7. **Adetoro, A.O.**, Fawole, O.A., Opara, U.L., Effect of harvest maturity on the physicochemical properties, phenolic content and antioxidant capacity of dried pomegranate (*Punica granatum*) arils (cv. Wonderful). *Agronomy* (under review).



## NOTE

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This dissertation presents a compilation of manuscripts where every chapter is an individual entity and some duplication between chapters, therefore, has been unavoidable.

## ACKNOWLEDGEMENTS

I would like to express my deep and sincere gratitude to the following people and organisations for their great contributions:

Prof Olaniyi Amos Fawole, my promoter, ‘this is a rare friend who brought me out to limelight’. I really appreciate his patience and inputs, especially during the postharvest discussion (PDF) forum. Thank you very much for all your support and feedback on my work.

Distinguished Professor Umezuruike Linus Opara, my co-promoter, South African Research Chair in Postharvest Technology (SARChI), for his support, constructive critique and encouragement. Thank you is not sufficient, but it is said with appreciation and great honour.

For financial support, I would like to thank The World Academy of Science and the National Research Foundation (NRF).

Prof M. Manley, Dr Oluwafemi Caleb, Dr Ambaw Tsige and Ms Nazneen Ebrahim, Postharvest Technology Research Lab, for their valuable input and assistance in assuring smooth running of the project. Also, a special thanks to my friends and postgraduate colleagues at SARChI Postharvest Technology Research Laboratory for their friendliness, encouragement, and input throughout my studies.

My parents, right (Adetoro), left (Ige-Olumide) and centre (CEDAR) I am thankful. You have always had confidence in my ability to succeed and have supported me in all my professional and academic endeavours. My wife Dr Olusola Olufayo Ige-Adetoro and my sons Iseoluwanimi Praise and Oreoluwanimi Zion Adetoro ‘You guys made this happen’ I am deeply thankful to you for the spiritual and moral support. Olusola, you are just my world.

Finally, my absolute thanks go to God through Jesus Christ, who gave me the grace to enjoy sound health, His help and good success throughout my studies.

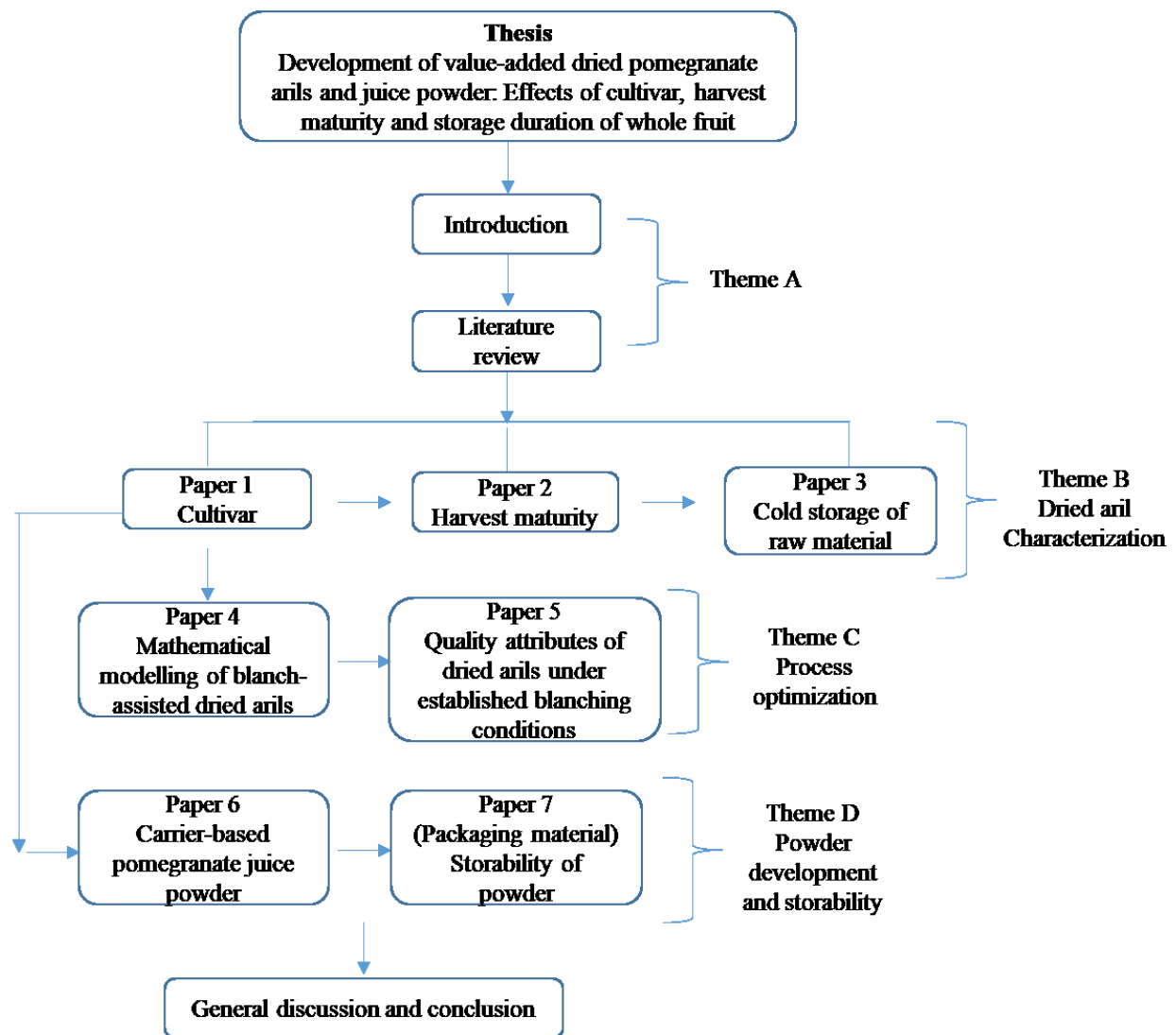
This work is based on the research supported wholly / in part by the National Research Foundation of South Africa (Grant Numbers: 64813). The opinions, findings and conclusions or recommendations expressed are those of the author(s) alone, and the NRF accepts no liability whatsoever in this regard.

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## Thesis structure flowchart



## **THEME A**

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- General Introduction
  - Thermal and non-thermal processing technologies of dried fruit products: pre- and postharvest implications on the quality attributes
-

# GENERAL INTRODUCTION

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## 1. Background

Global production of pomegranate (*Punica granatum*) has recently increased remarkably due to the potential health benefits of the fruit (Zeweil et al., 2013; Kalaycıoglu and Erim 2017). It has been regarded as a “healing food” with numerous beneficial effects on several diseases (Vidal et al., 2003; Tanveer et al., 2015). Pomegranate fruit was commonly used in folk medicine for eliminating parasites, as an anthelmintic and vermifuge, and to treat and cure aphthae, ulcers, diarrhoea, acidosis, dysentery, haemorrhage and microbial infections (Larrosa et al., 2010; Viuda-Martos et al., 2010). The health benefits of consuming pomegranate juice have been attributed to the exceptionally high antioxidant capacity that has been strongly linked to the high concentration and unique composition of fruit phenolic compounds (Gil et al., 2000; Fischer et al., 2011; Fawole and Opara, 2012). Epidemiological studies have stressed the importance of consumption of foods rich in phytochemicals such as fruit and vegetables in the prevention of non-communicable diseases (Opara et al., 2009; Fawole et al., 2012; Miller and Snyder 2012; Ruiz et al., 2016). These phytochemicals act as antioxidants and enhance immunity through their anti-carcinogenic, anti-microbial and anti-inflammatory effects, leading to a reduction in chemical biomarkers of health such as blood sugar and cholesterol (Liu, 2004; Fawole et al., 2012; Seymour et al., 2013; Arendse et al., 2014).

Pomegranates have now become an important commercial crop cultivated in different parts of the world, including South Africa (Fawole and Opara, 2012). With about 1000 ha of established pomegranate orchards and annual export volume of 540,000 tonnes, South Africa is a major producer of pomegranate in the Southern Hemisphere, competing with countries like Argentina, Peru and Chile (POMASA, 2018). The Western Cape Province, the pomegranate basket of South Africa, accounts for more than 80% of total production. The most important cultivars are Wonderful (70%), Acco (10%), Herskawitz (13%) and Kesari (2%) (POMASA, 2019). South African pomegranates are sold as whole fruit, juice or arils (Brodie, 2009). The fruit is comprised of two main parts, the peel (outer skin) and arils. Each aril contains juice and a kernel or seed. Arils are arranged in three or more sac-like structures, and constitute between 50-70% of whole fruit mass, while the kernel accounts for 15% of aril mass (Ghasemnezhad et al., 2015). However,

the shelf-life of pomegranate arils is short, usually between 5-7 days (Caleb et al., 2013). This results in high postharvest losses in the agro-processing sector of the industry, impacting the supply and availability of minimally processed pomegranate fruit. There is, therefore, a dire need to reduce postharvest losses in the agro-processing sector through the development and marketing of new products that are both nutritious and more shelf-stable.

Furthermore, low grade fruit such as pomegranate fruit with sunburn and cracks results in lower premium value in the market (POMASA, 2018). The conversion of low-grade fruit as raw materials for agro-processing is a strategy to derive high-value products, reduce wastage and yield more income for growers. Amongst the numerous methods for processing high moisture content of agricultural and horticultural food materials, drying is the most common method, since water removal suppresses the growth of microbes and reduces enzyme activity, thereby reducing spoilage, food loss and waste. Drying extends the shelf-life and decreases the weight of products, contributing to easy transportation and storage (de Bruijn et al., 2016). For the production of dried pomegranate products, various drying methods could be used, such as freeze-drying and hot-air drying, each having both advantages and disadvantages. For example, freeze-drying has been reported to significantly reduce browning and preserve the redness of dried pumpkin (Que et al., 2008). This process has been reported for its high retention of nutrients and flavourings in orange powder (Barbosa et al., 2015). Also, freeze dried products have high rehydration capacity (Tsami et al., 1999). However, freeze-drying is time consuming and expensive (Castro et al., 1997; Knorr, 1998). Hot-air drying allows exposure of solid materials to a continuous flow of convective hot-air, thereby evaporating the moisture content (Ratti, 2001). This process has been reported for its short drying time to obtain dried products with extended shelf-life (Flink, 1977) and enhancing the antioxidant capacity of dried pomegranate arils (Ozcan et al., 2018). In contrast, to freeze-drying, a drastic reduction in product quality with deformation and colour change is mostly associated with hot-air drying. Poor rehydration capacity of products obtained from hot-air drying has been reported (Ratti, 2001).

Studies on other types of fruit have established the effects of pre-harvest factors such as cultivar and harvest maturity on the quality of dried fruit products. For instance, sensory characteristics, phytochemical and antioxidant attributes of dried fruit can be influenced by cultivar differences (Pott et al., 2003; Haug et al., 2013). For three date cultivars investigated on



antioxidant capacity, as Al Farsi et al. (2005) reported that ‘Khalas’ had higher antioxidant activity, total carotenoids, and membrane bound phenolic acids than Fard and Khasab cultivars. According to Wojdyło et al. (2009), antioxidant capacity varied markedly depending on strawberry cultivar.

Similarly, the quality of dried fruit is also influenced by harvest dates or maturity. This factor mostly indicates the quality assessment of fruit to meet market requirement. For instance, less mature apples had low soluble solid concentration and dry matter than the medium and more matured samples (Rizzolo et al., 2011). Also, significantly higher sugar content in ripe (yellow) than unripe (green) banana was reported by Nguyen and Price (2006).

Furthermore, the optimum storage condition recommended for pomegranates is 5°C and >85% relative humidity, with a storage life of up to 3 months (Fawole and Opara, 2013). Studies have also shown that storage time influences the quality characteristics of the dried product in terms of colour, taste and aroma (Konopacka et al., 2001). For example, Rizzolo et al. (2011) reported that 3 months of dried apple ring storage had the highest differences for browning and total colour change compared to 0 and 5 months, while firmness decreased as the months increased. The perceptibility of taste and aroma, as well as colour change in dried products, are strongly dependent on the development of these characteristics in stored fruit. These findings highlight the need to study specific cultivars for commercial purposes, relevant harvest maturity indices and storage periods for optimal postharvest performance and sensory quality.

To add value and increase the availability of fruit-based products for consumers, good understanding of the influence of fruit cultivar, maturity status and postharvest storage conditions on a dried product is a necessity. Similarly, a better understanding of the nutritional and biochemical changes that occur during and after processing and storage of product is necessary to assist in identifying the exact threshold that relates to storability performance of the product. The above dynamics in the quality attributes of dried products as influenced by cultivar, harvest maturity and storage duration justify the need to carefully optimise the processing conditions to maintain quality and shelf-stability of value-added products from pomegranate fruit.

## 2. Aim and Objectives

### 2.1. *Aim*

The overall aim of this study was to develop value-added and shelf-stable dried arils and juice powder from pomegranate fruit.

### 2.2. *Objectives*

The specific objectives of this study were to:

- a. Assess the effect of fruit cultivar, harvest maturity and storage duration on quality attributes of dried pomegranate aril
- b. Investigate the effects of pretreatment and drying methods on quality attributes of pomegranate arils
- c. Establish suitable carriers for the development of pomegranate powder using freeze-drying method
- d. Study the storability and predict the shelf-life of freeze-dried powder

## 3. Thesis structure

This dissertation is structured into four themes (A - D), with each theme addressing a research focus.

- Theme A: provides a brief background and discusses the research aims and objectives (General introduction). It also provides a detailed review of current knowledge on thermal and non-thermal processing technologies of dried fruit products: pre- and postharvest implications on the quality attributes.
- Theme B: reports the studies on the characterization of pomegranate cultivars, harvest maturity and storage of whole fruit as influenced by drying as well as quality evaluation of the product
- Theme C: focuses on the process optimization of blanch-assisted dried arils concerning their quality attributes.
- Theme D: discusses the pomegranate juice powder development and storability.

The general discussion integrates the results of all the research themes. It also highlights the practical contribution of the studies to the South African pomegranate industry.

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## **Literature Review**

### **Thermal and non-thermal processing technologies of dried fruit products: pre- and postharvest implications on the quality attributes**

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#### **Abstract**

The increasing demand for safe and nutritious fruit and fruit products by consumers has promoted the rapid utilization of novel processing technologies. This review summarizes the influence of thermal and non-thermal processing technologies on the quality attributes of fruit products, including hot-air and freeze-drying technologies. These technologies have shown great potential for drying, concentration and deacidification. Processing technologies decrease processing time and temperature, improve processing efficiency, and minimize nutritional losses, as well as reduce energy consumption. Given the nutritional benefits of bioactive components in fruit, the effect of these technologies on dried products are also driven, in part, by the pre- and postharvest factors such as cultivar, harvest maturity and storage of raw materials. This review detailed the co-influence of processing technologies and the above-mentioned pre and postharvest factors on the quality attributes of dried fruit products. The underlying mechanisms were also highlighted.

**Keywords:** Quality, pretreatments, drying, cultivar, maturity, storage, raw material

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#### **1. Introduction**

Fruit are very important sources of essential nutritional diets such as vitamins, minerals and fibre. The consumption of fruits has been associated with reduced mortality rate and low incidences of several cancer types and heart diseases (Ames et al., 1993). They are known to boost the immune system, detoxify contaminants, and reduce inflammation (Andersen and Jordheim 2006). These are greatly attributed to the phenolic compound, antioxidant activities, fibres, vitamins and mineral contents present in fruit (Andersen and Jordheim 2006; Siriamornpun et al., 2012). In a large extent, these chemical compounds are referred to as chelate metal ions whereby scavenging harmful free radicals relates with acute and chronic diseases in order to protect human internal organs and systems from oxidative damages (Edge et al., 1997; Heim et al., 2002; Johnson 2002; Balasundram et al., 2006; Karam et al., 2016).

However, since the moisture content of fresh fruits is up to 80 %, it renders them to be highly perishable (Orsat et al., 2006). Maintaining the product as fresh as possible is the best way

to keep its nutritional value, whereas, most storage methods demands low temperatures, which are usually difficult to maintain for all the periods of distribution. Hence, drying is a suitable alternative technology for the postharvest management of fruits.

Drying is a process whereby water is withdrawn from a food product to stop the growth of microorganisms and nutrient deterioration in the material matrix (Zhang et al., 2006; Argyropoulos et al., 2011; Kurozawa et al., 2012), hence, retaining as much as possible the quality and shelf-life of the product (Aguilera 2003). Many centuries back, drying through the means of sun and solar drying techniques have been engaged to be one of the most used preservation methods for fruits and vegetables (Van Arsdel et al., 1973). Alternatively, several drying technologies have been developed to alleviate the use of sun drying method further basically to avoid product contamination, especially in the temperate region. The recently developed drying technologies include microwave, ultrasound, freeze, spouted bed and the use of combined methods (George et al., 2004), spray drying (Muzaffar et al., 2015), refractance window drying (Caparino et al., 2012). Apart from freeze-drying, irradiation drying and ultra-high-pressure processing which are considered as non-thermal dryers due to their low temperature operations (Onwude et al., 2017). Heat applied on products during the process of drying are through conduction, convection and radiation, which are ways of forcing water into vapour while forced air is applied. The selection of a drying method is determined by several factors, including the product type, quality of the final product and economic value in terms of cost, time and energy consumption.

Globally, drying accounts for 10-25% of the total energy being utilized during the food manufacturing process, which makes it an energy-intensive process (Strumillo and Adamiec 1996; Mujumdar 2007; Ahmed 2011). Fruits are dried to improve shelf-life, reduce packaging costs, lower shipping weights, enhance appearance, maintain original flavour and nutritional composition. Several processing technologies have been engaged for the drying of fruits. Hot-air drying has primarily been utilized to achieve drying of various fruits (Nijhuis et al., 1998). Several studies have shown several limitations associated with conventional convective drying. Changes in some physical properties such as colour (Chua et al., 2000), texture, total soluble solids, titratable acidity and shrinkage have observed (Mayor and Sereno 2005). Also, one of the causes of quality loss is drying at a high temperature. However, the minimum temperature during drying has a greater tendency to improve the quality of dried products (Nindo et al., 2003; Beaudry et al., 2004). It provides a relatively slow, uniform and sanitary drying which however contributes to

economic loss due to elongation of operating time and associated cost. Other causes of quality loss are discolouration due to enzymatic browning (Thakur et al., 2010). Enzymes responsible for these activities include polyphenol oxidase and peroxidase (Meighani et al., 2014).

However, in order to minimize the rate of degeneration of products during drying, a thermal process referred to as heat pretreatment is essential. Several studies have shown that heat pretreatments mainly reduce the level of quality deterioration in fruit during and after drying. Another advantage of heat pretreatment is the reduction of operational cost, in terms of energy consumption and duration. Overall, the choice of drying process plays a significant role in maintaining the quality potential of the dried fruit product. This review examines the influence of processing technologies as well as the pre- and postharvest factors on the quality attributes of dried fruit products.

## **2. Effect of heat pretreatment on quality of dried attributes of fruit products**

Heat pretreatments have been reported to suppress natural enzyme activity, accelerate the drying process and retain the quality of fresh and dried fruit products. These methods include blanching and steaming pretreatments (Table 1), and the effectiveness of each treatment depends on fruit type.

Maghoumi et al. (2013) investigated the effects of hot water dipping at 55 °C for 30 s on quality attributes of pomegranate arils and found that the treatment reduced superoxide dismutase activity (SOD) compared to control. This finding is in agreement with Thakur et al. (2010), who reported that steam blanching of arils for 30 s reduced drying time and reduced non-enzymatic browning. According to the author, dried arils had the maximum scores for sensory characteristics like colour, texture, taste, aroma and overall acceptability for the pre-treated arils. On mangosteen fruit hot water dipping in the range of 60-100 °C, Deylami et al. (2016) reported that anthocyanin content was the most sensitive parameter at all temperatures and blanching enhanced the efficiency of anthocyanin extraction. However, blanching as a single pretreatment method was not sufficient to inhibit enzyme activity. Similarly, Peerajit et al. (2012) worked on lime residues blanched in hot water at 95 °C for 5 min and subsequently soaked in ethanol solutions for 30 min. The authors reported that the dietary fibre powder prepared by blanching before drying exhibited higher glucose retardation index (GRI) and bile acid retardation index (BRI) (Table 1).



Similarly, Sablani et al. (2011) noted that the application of blanching in red raspberries and blueberries resulted in enhanced moisture transport, thus reducing the drying time. The authors also reported that blanching treatment before air drying aided the retention of phytochemicals in dried berries. Rodrigues et al. (2015) assessed the effect of blanching on yellow passion fruit and reported that blanching shortens the drying time of fruits. The physicochemical and technological properties were preserved due to blanching conditions (Table 1). Similarly, Nurhuda et al. (2013) established that for rambutan peel, both water and steam blanching for 0, 2.5 and 5 min in boiling water at 100 °C significantly reduced polyphenol oxidase (PPO) and peroxidase (POD) activities. Also, for antioxidant activity, the thermal pretreatment caused no significant difference in the contents of phenolic compounds, as well as the antioxidant capacity of the final product (Table 1).

### **3. Effects of non-thermal and thermal drying technologies on quality loss/ retention in dried fruit**

Several changes are observed in the nutritional quality of fruits during or after drying processes. Several factors determine the degree to which these changes occur; the amount of treatments the material is exposed to before drying, drying methods applied and the operating conditions such as temperature, humidity and velocity of the drying medium. Some of the quality parameters attributed to dried fruit products are colour, appearance, flavour, nutrient retention, texture, rehydration properties, water activity and the attractive scent of the product (Sagar and Suresh 2010). In addition to the quality of raw material, the choice of drying technologies also plays a significant role in quality retention or stability of the product (Santos and Silva 2008). The existing drying technologies can be classified as non-thermal and thermal. Non-thermal refers to the use of temperatures below standard room conditions, which includes temperatures below or close to the product's freezing point and high pressure (Ozuna et al., 2014; Onwude et al., 2017). In the case of the thermal drying, the application of energy in the form of heat is required to evaporate the liquid (Gallego-Jukez et al., 1999). This method eradicates moisture from the product.

#### **3.1 Non-thermal drying technology — freeze-drying**

Freeze drying is also known as lyophilisation. This drying method involves two drying processes—first, the sublimation process, which is regarded as the primary drying process and second is the process of desorption — the secondary process of drying in which water initially frozen is sublimed from the product without passing through the liquid state (Nireesha et al., 2013).

Freeze-drying is one of the most advanced drying techniques which provides dried products with porous structure (Mosquera et al., 2012). The material to be dried is first frozen and then subjected to high vacuum-conductive heat to attain only solidly dried products (Nireesha et al., 2013). It is a drying process that elongates the storage and shelf-life of products and promotes stability in the dry state (Lippincott and Remington 2000). It is widely known to retain flavour and aroma with negligible shrinkage and improves rehydration behaviour (Mosquera et al., 2012). However, the period of handling and processing is prolonged.

Table 2 describes the freeze-dried materials at an operating temperature ranging between 50 to -23 °C and time ranging between 24 to 72 h. Their characteristic qualities were described by the highest retention of bioactive compounds such as polyphenols, lycopene content, total flavonoids and anthocyanin content (Michalczyk et al., 2009; Asami et al., 2003; Chang et al., 2006; Sablani et al., 2011). High ascorbic acid retention of up to 96 % in star fruit, papaya, mango, strawberry, guava pulp and watermelon (Shofian et al., 2011; Mosquera et al., 2010; Shishehgarha 2002; Ferreira et al., 2004). Sensory quality and flavour such as sweeter taste, improved product appearance, colour retention and powder stability, wettability and water solubility in dried banana, borojo powder, soursop pulp and strawberry (Oikonomopoulou and Krokida 2013; Paakonen and Mattila 1991; Mosquera et al., 2010; Ceballos et al., 2012; Shishehgarha 2007).

Further, several researchers (Krokida et al., 2000; Mujaffar et al., 2015; Ceballos et al., 2012) studied the effects of drying methods (such as convective, vacuum, microwave, osmotic and freeze drying) on the colour of dehydrated fruit products (Table 2). The authors reported that freeze-dryer at operating condition (-20°C, -70°C) and time 72 h prevented discolouration, resulting in products with improved colour characteristics. Some authors have observed loss of structure, reduction in pore sizes and rate of collapse in strawberries with increase in drying temperature (Shishehgarha 2002; Hammani and Rene 1997). Even though freeze-drying results in more shelf-stable products, the equipment is expensive, and it requires prolonged drying time (Karam et al., 2016).

### **3.2. Thermal drying technology – hot-air drying**

This technique is usually referred to as air-drying or convective drying. It is the process of preserving food materials in continuous streamflow of hot-air until the moisture in the product evaporates (Ratti 2001). Because of its economic benefits, it is a widely adopted technique in the food industry. Zielinska and Michalska (2016) evaluated the effect of hot-air convective drying in

comparison with other driers, an effective response was noticed with hot-air drying as one of the driers mostly efficient in drying fruits. Fick's second law of diffusion equation is mostly used to describe moisture movement during drying (Karam et al., 2016). Alongside moisture loss, shrinkages and microstructural changes in dried fruits are associated with the reduction in moisture. Several authors have reported a severe shrinkage of up to 80% in berries, kiwi, apple slices and grape according to (Jankovic 1993; Maskan 2001; Mayor et al., 2005; Adiletta et al., 2016) respectively during air drying (Table 3). Furthermore, the loss of water causes a decrease in cell size, roundness and compactness as observed in apple slices (Mayor et al., 2005).

Further, convective dried products at processing conditions between (27 °C – 70 °C, at air velocity 2-3 m s<sup>-1</sup>) were characterized by shortened drying time, lower rehydration capacity and slower water absorption rate among other drying methods (Wang et al., 2007; Maskan 2001; Maskan et al., 2002; Adiletta et al., 2016; Krokida and Philippopoulos 2005) (Table 3). However, instability of colour during drying was observed in hot-air dried fruit, at processing condition ranging between (30 °C – 70 °C, at air velocity 3-5 m s<sup>-1</sup>), also with a decrease in lightness and increased yellowness values (Karam et al., 2016) as found for air-dried fruits (Sacilik and Elicin 2006; Mrad et al., 2012; Krokida et al., 2001; Tsami and Katsioti 2000; Krokida and Maroulis 1998; Karabulut et al., 2007; Wodjylo et al., 2014), and induction of browning pigments (Voegel-Turenne et al., 1997). A drastic loss of ascorbic acids varying from 25-91% was observed during hot-air drying for several fruits (Desrosiers et al., 1985; Ramesh et al., 2001; Di Scala and Crapiste 2008; Vega-Galvez et al., 2009; Mrad et al., 2012) (Table 3).

In addition, Krokida and Philippopoulos (2005) and Askari et al. (2009) reported low retention of flavour and aroma in apple, as well as a reduction in the quality of optical properties in apple, strawberry and tomatoes during hot-air drying with operating conditions (50–90 °C; 5 kW ) (Table 3). Regarding the phytochemical content, (Ramesh et al., 2001; Shi et al., 1999) have found that carotenoid compounds, more especially lycopene, were heat sensitive after severe heat treatment (60 °C air temperature and 1.0 ± 0.25 m/s air velocities, 10 ± 2% relative humidity). They have demonstrated that lycopene retention is reduced in hot-air dried apple, banana and guava. Moreover, some authors have reported that hot-air drying reduced the contents of total phenolics, antioxidants and anthocyanin concentration as observed in air-dried red pepper, apple, pear, grape, sour cherries, tomatoes and berries (Mrad et al., 2012; Wodjylo et al., 2014; Sablani

et al., 2011; Piga et al., 2004) (Table 3). Apart from the influence of hot-air drying on food quality, the process is most important in many sectors in the food industry mainly because of the increased shelf-life of the product, lower packaging cost, reduced shipping weights and environmental merits (Lewicki 2006).

#### **4. Effect of fruit cultivar on dried product quality**

The quality of fresh fruit varies significantly with several factors, including cultivar, maturity, and postharvest storage conditions (Prior et al., 2010). Studies have shown that quality attributes of dried fruit products derived from either thermal or non-thermal processing are influenced and vary among cultivars. Influence of cultivar differences on functional properties of dried fruit product is summarized in Table 4. Al-Farsi et al. (2005) investigated the antioxidant activity, anthocyanins, carotenoids, and phenolics of three native fresh and sun-dried date (*Phoenix dactylifera* L.) varieties grown in Oman. The authors reported that Khalas variety had higher antioxidant activity, total carotenoids, and bound phenolic acids than other varieties. Loss (ranging from 4 to 29.8%) of carotenoids in date varieties after drying was found in other cultivars except in Khalas which was insignificant. The mean total content of phenolics ranged from 217 to 343 mg of ferulic acid equivalent (FAE)/100 g in sun-dried date varieties.

Similarly, in Table 4, Wojdylo et al. (2009) found higher antioxidant activity in strawberry Elsanta cultivar than Kent. The Elsanta cultivar (1857.1 mg/100 g of dw) contained higher amounts of proanthocyanidins than Kent (1373.6 mg/100 g of dw). Total anthocyanin contents of strawberry fruits were 294.4 and 372.6 mg/100 g of dw for cultivars Kent and Elsanta, respectively. Given the differences in the cultivars, regardless of the processing method employed (thermal; vacuum-microwave drying at 240, 360, and 480 W and non-thermal; freeze drying), Elsanta, which had more polyphenolic compounds and vitamin C, had higher antioxidant activity than the Kent cultivar. Similarly, studies by Pott et al. (2003) observed total and partial degradation of xanthophylls and all-trans- $\alpha$ -carotene respectively for Kent and Tommy Atkins cultivars of dried mango. Values of vitamin A found in dried product in both cultivars were 752 retinol activity equivalents (RE)/100g and 431 RE/100 g, respectively (Table 4). Contents of  $\beta$ -carotene were 4270 and 2510  $\mu$ g/100 DW for Kent and Tommy Atkins cultivar, respectively. Ascorbic acid content between 3.57 and 2.48 mg/100g was reported by Madrau et al. (2009) for Cafona and Pelese cultivars of dried apricot (air temperature 55 and 75 °C) (Table 4). Further, the authors found total phenols for Cafona and Pelese as 600 and 50 mg/kg/Dm. The antioxidant activity was

also reported to be high in Cafona 34  $\text{OD}^{-3} \text{min}^{-1} \text{g}^{-1} \text{dm}^{-1}$  compared to Pelese with 5  $\text{OD}^{-3} \text{min}^{-1} \text{g}^{-1} \text{dm}^{-1}$ .

Haug et al. (2013) found that all cultivars of dried figs at 20 °C processed condition, have predominant sensory characteristics for Black Mission, Calimyrna, Conadria, Kadota, Sierra, and Tena varieties grown in USA. Conversely, Demirel and Turhan (2003) observed that the extent of the change in the physical properties was higher in the Dwarf cavendish than in the Gros michel dried banana slices investigated between 40 and 70 °C. They also found that Cavendish samples shrank more than Michel samples at the same drying conditions. Also, in Table 4, Wojdylo et al. (2016) studied the chemical composition, antioxidant capacity, and sensory quality of dried jujube fruits as affected by cultivar and drying method; convective drying (CD: 50, 60, 70° C), vacuum-microwave drying (VMD: 120, 480, 480-120 W). The authors reported that the best cultivars were “PSI” and “GAL” based on the bioactive content and sensory quality, respectively. Furthermore, the authors found contents of flavan-3-ols and flavonols were found at the highest level in “PSI” fruits obtained using the control drying method, FD (4297 mg/100 g dm), followed by “MSI” and “GAL”, with contents of 3306 and 3007 mg/100 g dm, respectively. Also, the highest content of vitamin C of all drying treatments, with values ranging from 2160 mg/100 g dm in “GAL” fruits (387 mg/100 g fresh mass, FM) to 3558 mg/100 g dm in “PSI” fruits (554 mg/100 g FM). “PSI” had the highest ABTS and FRAP, followed by “MSI” and “GAL” fruits. “GAL” fruits had a significantly lower intensity of skin colour than fruits of the other two cultivars (“MSI” and “PSI”); “GAL” fruits kept a yellow-green colour while the other dried fruits had a significantly darker colour (Table 4).

## 5. Effects of fruit harvest maturity on dried product quality

Studies have shown that chemical properties of fruit are mostly dependent on developmental and ripening stages (Toor et al., 2006; Mphahlele et al., 2014) which plays a significant role in determining the quality composition of fruit. However, very little information has been reported on the effect of maturity status on the quality of dried fruit products. The few information was reported on thermal processing without any information on non-thermal processing effect on the harvest maturity of dried fruit. A summary of the effects of harvest maturity status on quality attributes of dried fruit products is presented in Table 5. Rizzolo et al. (2011) investigated the quality characteristics of apple rings air-dried at 80 °C and stored in normal atmosphere (+1 °C) for up to 5 months.

The authors reported that fruit maturity level influenced the mechanical characteristics of dried apple rings. Ring hardness ( $F_{\max}$ ) and the energy-to-breakpoint ( $E$ ) were higher in rings from fruit processed at harvest and those of less matured class. Apple rings from less matured fruit also showed higher redness than other maturity rings. However, rings from less mature fruit had lower soluble solids and dry matter compared with those of more maturity fruit. Similarly, in Table 5 a study on air-drying of banana as influenced by harvest maturity was investigated at 10 °C intervals between 30 and 70 °C and 1 m/s air velocity (Nguyen and Price 2006). The authors reported a significant rise in the amount of sugar in yellow-ripe than a green banana. Furthermore, mass loss between green and ripe banana was very similar.

## **6. Effects of storage of raw materials on dried product quality**

Fruit aimed for formulation of new products or fortification of existing product are rarely processed immediately after harvest because of several factors such as the choice to permit further ripening of the fruit to process products with specific desired attributes, the necessity to exhaust previously harvested fruit to avoid excessive spoilage, or simply the need to elongate the time of processing (McLellan and Massey, 1984). Given that the quality of the final product is prepared from raw material (whole fruit), most postharvest quality of the raw material degrades over time during storage. However, there is a dearth of information on the effect of prolonged storage of raw material on the quality of processed products. The only articles on this crucial aspect of the processing of dried fruit products were those by McLellan and Massey (1984); Hsu et al. (1989) and Konopacka and Plochanski (2001).

McLellan and Massey (1984) reported the sensory quality of processed applesauce as influenced by postharvest storage and ripening at 0 °C and 18 °C storage for 10 weeks. According to the authors, pre-processing low temperature storage of apples increased the perceived sweetness and decreased sourness (tartness) of the finished applesauce. Similarly, Hsu et al. (1989) reported that total soluble proteins decreased with storage time in apple juice stored at 1 °C for three months and nine months. Also, juices processed from three months storage were more resistant to heat stability test than those processed from nine months of storage. In the same vein, the variations of dried chips' sensory quality were reported to be influenced by the biochemical changes naturally occurring in ripening stored apples under air storage at 0 °C for 6 months (Konopacka and Plochanski, 2001).

## 7. Future prospects and conclusion

Recently, the development of shelf-stable products with optimal quality retention of dried fruit has been a subject of interest among researchers due to the seasonal availability of fruit and the urgent need to the current high incidence of losses and waste in the horticultural sector. In this article, an overview was presented on recent findings on the effects of thermal and non-thermal processing on the quality attributes of dried fruit products. Furthermore, pre- and postharvest factors affecting dried fruit were presented including cultivar, harvest maturity and storage duration. Available evidence has shown that considerable loss of nutrients such as phenolic content and antioxidant capacity occurs during drying; however, overall quality retention is mostly dependent on the operating conditions during processing. The application of heat pretreatment was identified as a plausible method to reduce such sensory and nutritional quality losses in processed products.

It is noteworthy that very few studies in literature reported little information on the effects of cultivar differences and harvest maturity on the quality of dried products. Future studies should focus on the influence of the storability of raw material on quality attributes of processed/finished products. In addition, the scope for research on the application of modelling to optimise drying processes for different fruit cultivars and harvest maturities to improve the understanding of the mechanism of action responsible for various beneficial effects of dried fruit is necessary. These new research directives will assist in the development of improved handling and processing protocols for the production and supply of novel products with consistent good quality, tasty, flavourful and healthful dried products to consumers.

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**Table 1**  
**Effect of heat pretreatment on the quality attributes of dried fruit products**

Heat methods	Fruit(s)	Methodology	Key findings	Reference
Hot water dipping	Pomegranate arils	Fresh-cut arils were subjected to hot water dipping at 55 °C for 30 s	<ul style="list-style-type: none"> <li>• Treatment reduced superoxide dismutase activity.</li> <li>• Hot water was effective suppressing polyphenol oxidase activity while peroxidase activity increased.</li> </ul>	Maghoumi et al. (2013)
Steam blanching	Wild pomegranate arils	After blanching, arils were fumigated in a chamber by burning sulphur for varying time at ambient temperature (25 °C).	<ul style="list-style-type: none"> <li>• Steam blanching of arils for 30 s and sulphur fumigation at 0.3 % for 60 min was found suitable</li> <li>• Minimum drying time</li> <li>• Minimum non-enzymatic browning and moisture contents.</li> <li>• The dried arils recorded maximum scores for sensory characteristics like colour, texture, taste, aroma and overall acceptability for the standardized pretreatment.</li> </ul>	Thakur et al. (2010)
Blanching	Mangosteen pericarp	This was dried out over a temperature range of 60-100 °C.	<ul style="list-style-type: none"> <li>• Anthocyanin content was the most sensitive parameter towards temperature changes</li> <li>• Blanching enhanced the efficiency of anthocyanin extraction</li> <li>• Blanching as a single pretreatment method was not sufficient to inhibit enzyme activity</li> </ul>	Deylami et al. (2016)
Heat methods	Fruit(s)	Methodology	Key findings	Reference

Blanching	Lime	Lime residues (600 g) were blanched in hot water at 95 °C for 5 min and subsequently soaked in ethanol solutions for 30 min.	<ul style="list-style-type: none"> <li>• DF powder prepared either by blanching or blanching followed by ethanol soaking prior to drying exhibited higher GRI and BRI.</li> <li>• DF powder with smaller sizes had higher GRI and BRI.</li> </ul>	Peerajit et al. (2012)
Blanching	Red raspberries and blueberries	Berries were steam blanched at 95 °C for 2 min and then cooled to 38 °C.	<ul style="list-style-type: none"> <li>• Blanching resulted in enhanced moisture transport, thus reducing the drying time.</li> <li>• The blanching treatment prior to air drying increased the retention of phytochemicals in dried berries.</li> </ul>	Sablani et al. (2011)
Blanching and hot-air drying	Yellow passion fruit	Samples were blanched at 90 °C for 1 min (skin-water ratio was 1:3) and then the samples were immersed in cold water (48 °C) for 5 min.	<ul style="list-style-type: none"> <li>• Shortest drying time (2.5 h) was obtained at 60 °C and 2.7 m/s for both control and blanched fruits.</li> <li>• The physicochemical and technological properties were not affected by the drying conditions</li> <li>• Physicochemical and technological properties were not affected by blanching</li> </ul>	Duarte et al. (2016)
Blanching	Rambutan	Rambutan peels were blanched for 0, 2.5 and 5 min in boiling water at 100 °C. After blanching, the samples were immediately cooled in an ice bath to stop the heating process.	<ul style="list-style-type: none"> <li>• Both water and steam blanching significantly reduced POD and PPO activities.</li> <li>• Increase in the blanching period did not significantly reduce the enzyme activities further.</li> <li>• Thermal pretreatment caused no significant difference in the phenolic contents and the antioxidant capacity of the final product.</li> </ul>	Nurhuda et al. (2013)

**Table 2**  
**Quality attributes of dried fruit products obtained by freeze-drying**

Product	Processing condition	Remarks	References
Raspberry, strawberry and bilberry	-23 °C 24 h	<ul style="list-style-type: none"> <li>Retained high polyphenol and anthocyanin content and high antioxidant</li> </ul>	Michalczyk et al. (2008)
Marionberry, strawberry	-32 °C	<ul style="list-style-type: none"> <li>Preserved total phenolics and ascorbic acid</li> </ul>	Asami et al. (2003)
Borojo powder	-40 °C 48 h	<ul style="list-style-type: none"> <li>Improve powder stability</li> <li>Decrease hygroscopicity</li> </ul>	Mosquera et al. (2010)
Apple, Banana	-35 °C for 48 h	<ul style="list-style-type: none"> <li>Retained colour better than other drying methods</li> </ul>	Krokida et al. (2001)
Tomatoes	-50 °C, 5 pa, for 24 h	<ul style="list-style-type: none"> <li>Increasing total flavonoids, total phenolics, lycopene contents</li> </ul>	Chang et al. (2006)
Banana	-30 °C for 72 h	<ul style="list-style-type: none"> <li>High porosity due to lowest temperature/ More intense shrinkage and sweeter taste observed</li> </ul>	Oikonomopoulou and Krokida (2013)
Organic berries	-30 °C for 48 h	<ul style="list-style-type: none"> <li>Improved water potential</li> <li>Increase in total phenolics and concentration of anthocyanin</li> </ul>	Sablani et al. (2011)
Star-fruit, papaya, muskmelon and mango and watermelon	-	<ul style="list-style-type: none"> <li>Almost complete carotene retention (96 %) was observed</li> <li>Decrease in B-carotene concentration was observed</li> <li>High ascorbic acid retention</li> <li>Increased antioxidant activity</li> </ul>	Shofian et al. (2011)

Product	Processing condition	Remarks	References
Apple, Banana	-35 °C for 48 h	<ul style="list-style-type: none"> <li>Retained colour better than other drying methods</li> </ul>	Krokida et al. (2001)
Apple	-20 °C and -80 °C for 49.5 h and 50.5 h respectively	<ul style="list-style-type: none"> <li>Texture losses of rehydrated apple were about 85 %</li> </ul>	Hammami et al. (1999)
Pumpkin	-44 °C for 64 h	<ul style="list-style-type: none"> <li>High colour retention was observed in product</li> </ul>	Mujaffar et al. (2015)
Sapota powder	-40 °C	<ul style="list-style-type: none"> <li>Brighter colour, high rehydration ability</li> </ul>	Jangam et al. (2008)
Pineapple, barbados cherry, guava, papaya and mango	-30 °C	<ul style="list-style-type: none"> <li>Flavour and taste of product were preserved</li> <li>Colour was retained</li> <li>Vitamin C, Calcium and Phosphorus were conserved</li> </ul>	Marques et al. (2006)
Strawberry	-20 °C and -60 °C	<ul style="list-style-type: none"> <li>Improved sensory quality of dried strawberries</li> </ul>	Paakonen and Mattila (1991)
Strawberry	-20 °C and -80 °C	<ul style="list-style-type: none"> <li>Colour and other nutritional attributes were enhanced</li> <li>Loss of structure, reduction in pore size</li> </ul>	Hammani and Rene (1997)
Soursop fruit pulp	-44 °C for 6 h	<ul style="list-style-type: none"> <li>Increased wettability and water solubility.</li> <li>Lighter colour observed</li> </ul>	Ceballos et al. (2012)
Marionberry, strawberry	-45 °C for 20-24 h	<ul style="list-style-type: none"> <li>No differences in total phenolics compared to fresh samples.</li> <li>Retention of ascorbic acid</li> </ul>	Asami et al. (2003)



**TABLE 3****Quality attributes of dried fruit products obtained by hot-air drying**

Product	Processing condition	Remarks	References
Green pepper and peach	32-62 °C, blanching 98 °C for 2 min	<ul style="list-style-type: none"> <li>Loss of ascorbic acids are 25 and 75 % in green peppers and peaches</li> </ul>	Desrosiers et al. (1985)
Paprika	60 °C air temperature and 1.0 ± 0.25 m/s air velocities, 10 ± 2% relative humidity	<ul style="list-style-type: none"> <li>Carotenoid retention is more in pre-treated product</li> <li>Considerable loss of vitamin C due to drying 75-91%</li> </ul>	Ramesh et al. (2001)
Apple slices	Drying temperature: 40, 50, and 60 °C. thickness: 5 to 9 mm	<ul style="list-style-type: none"> <li>Untreated sample and lower temperature maintained original colour of fresh apple slices better as compared to treated one</li> </ul>	Sacilik and Elicin (2006)
Strawberry, raspberry and blackberry	-	<ul style="list-style-type: none"> <li>Up to 80% shrinkage of product</li> </ul>	Jankovic (1993)
Kiwi	60 °C dry bulb and 27°C wet bulb	<ul style="list-style-type: none"> <li>Less shrinkage observed at hot-air</li> <li>Lower rehydration capacity and slower water absorption rate</li> </ul>	Maskan (2001).
Red pepper	Inlet temperatures 50, 60, 70, 80 and 90 °C	<ul style="list-style-type: none"> <li>Vitamin C content and the total phenolic content decreased as air-drying temperature decreased</li> <li>Higher antioxidant activity at high temperatures</li> </ul>	Vega-Galvez et al. (2009)
Grape	40–70 °C and at 2.3 ms <sup>-1</sup> air velocity	<ul style="list-style-type: none"> <li>improved rehydration capability</li> <li>Preserved the antioxidant activity and reduced the shrinkage of grapes</li> </ul>	Adiletta et al. (2016)

Product	Processing condition	Remarks	References
Apple slices	70 °C	<ul style="list-style-type: none"> <li>Decrease in cell size, roundness and compactness</li> </ul>	Mayor et al. (2005)
Pineapple slices	30–60 °C	<ul style="list-style-type: none"> <li>Decrease in ascorbic acid</li> </ul>	Orikasa et al. (2010)
Red pepper	50–70 °C Air velocity 0.2–1.2 m/s	<ul style="list-style-type: none"> <li>Ascorbic acid was degraded with high temperature</li> </ul>	Di Scala and Crapiste (2008)
Apple	30–70 °C	<ul style="list-style-type: none"> <li>Low retention of flavour and aroma</li> </ul>	Krokida and Philippopoulos (2006)
Pear	30–70 °C	<ul style="list-style-type: none"> <li>Degradation in ascorbic acid and total phenolic content with high temperature</li> <li>Porosity higher at highest temperatures (&gt;50 °C)</li> <li>Greater change in colour with high temperature</li> </ul>	Mrad et al. (2012)
Grape juice and leather	40–90 °C Wet bulb temp.: 27-33 °C	<ul style="list-style-type: none"> <li>Reduced drying time than Sun drying</li> </ul>	Maskan et al. (2002)
Apple, banana	70 ± 0.2 °C and 7% air-relative humidity, pressure at 1 bar ± 3 %.	<ul style="list-style-type: none"> <li>Decrease in colour retention</li> </ul>	Krokida et al. (2001)
Avocado, prune, strawberry	50–70 °C air velocity: 3–5 m/s	<ul style="list-style-type: none"> <li>Color of avocado and strawberry changes while the color of the prune remains the same</li> </ul>	Tsami and Katsioti (2000)
Apple, banana	50–90 °C	<ul style="list-style-type: none"> <li>a* and b* depends on temperature and humidity</li> </ul>	Krokida and Maroulis (1998)
Apple	40–90 °C	<ul style="list-style-type: none"> <li>Induction of browning pigments depends on time</li> </ul>	Voegel-Turenne et al. (1997)

Product	Processing condition	Remarks	References
Apricots	50, 60, 70, and 80 °C	<ul style="list-style-type: none"> <li>• Colour deterioration due to Maillard reactions</li> <li>• Remarkable decrease in lightness and increase in yellowness values</li> </ul>	Karabulut et al. (2007)
Banana, apple, pepper, tomato and pumpkin.	-	<ul style="list-style-type: none"> <li>• Irreversible structural changes during drying leads to the inability of air-dried tissues to imbibe sufficient water for full rehydration</li> </ul>	Krokida and Philippopoulos (2005)
Sour cherries	50 °C, 60 °C, and 70 °C	<ul style="list-style-type: none"> <li>• Increase in air temperature deteriorated dried product quality in terms of the content of phenolic compounds, antioxidant activity, and color, which was consistent with anthocyanins content</li> </ul>	Wodjylo et al. (2014)
Tomatoes	95 °C for 6–10 h	<ul style="list-style-type: none"> <li>• Reduction in the content of lycopene</li> </ul>	Shi et al. (1999)
Blueberry, red raspberries	Air inlet temperature of 65 °C	<ul style="list-style-type: none"> <li>• Increase in antioxidants and phenolic concentration</li> <li>• Decrease in anthocyanins</li> </ul>	Sablani et al. (2011)
Apple, strawberry, tomato	Electric heater rated at 5 kW	<ul style="list-style-type: none"> <li>• Reduction in the quality of optical properties of dried materials</li> </ul>	Askari et al. (2009)
Plums	85–90 °C for 18 h	<ul style="list-style-type: none"> <li>• Increase or decrease in polyphenol oxidase or non-oxidase after hot-air drying is ascribed to the substrate nature</li> </ul>	Piga et al. (2003)

**Table 4****Effect of cultivar differences on the quality of dried fruit product**

Product	Cultivar	Country	Fruit part/fraction	Key findings	References
Date	Fard, Khasab & Khalas	Oman	Whole	<ul style="list-style-type: none"> <li>• Khalas had higher antioxidant activity, total carotenoids, and bound phenolic acids than other varieties.</li> </ul>	Al-Farsi et al. (2005)
Figs	Black Mission, Calimyrna, Conadria, Kadota, Sierra, and Tena	USA	Whole	<ul style="list-style-type: none"> <li>• All cultivars had predominant sensory characteristics.</li> </ul>	Haug et al. (2013)
Mangoes	Kent and Tommy Atkins	Germany	Slices	<ul style="list-style-type: none"> <li>• Drying resulted in a complete and partial degradation of xanthophylls and all-trans-<math>\alpha</math>-carotene respectively for both cultivars</li> </ul>	Pott et al. (2003)
Strawberry	Kent and Elsanta	Poland	Whole	<ul style="list-style-type: none"> <li>• A higher antioxidant activity was observed for Elsanta than Kent.</li> <li>• Radical scavenging activity was higher in Kent than Elsanta</li> </ul>	Wojdyło et al. (2009)
Apricots	Pelese and Cafona		Whole	<ul style="list-style-type: none"> <li>• Ascorbic acid increased significantly in Cafona than Pelese</li> </ul>	Madrau et al. (2009)
Banana	Dwarf Cavendish and Gros Michel	Turkey	Slices	<ul style="list-style-type: none"> <li>• Extent of the change in the physical properties was more pronounced in the Cavendish banana slices than in the Michel banana slices</li> </ul>	Demirel and Turhan (2003)
Jujube fruit	GAL, MSI and PSI	Poland	Slices	<ul style="list-style-type: none"> <li>• Best cultivars were PSI and GAL for bioactive content and sensory quality</li> </ul>	Wojdyło et al. (2016)

**Table 5****Effect of harvest maturity on the quality of dried fruit product**

Product	Maturity status	Country	Fruit part/Fraction	Key findings	References
Apple	Less mature, medium mature & more	Italy	Rings	<ul style="list-style-type: none"> <li>• Less mature apples had low soluble solids content and dry matter than two other samples.</li> <li>• Improved colour was found in the more matured sample and high browning index was observed for less mature samples.</li> </ul>	Rizzolo et al. (2011)
Banana	Ripe and green maturity	Australia	Slabs	<ul style="list-style-type: none"> <li>• Mass loss between green and ripe banana was very similar</li> <li>• Significant rise in the amount of sugar in ripe banana than green</li> </ul>	Nguyen and Price (2006)

## THEME B

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### ARIL PROCESSING

#### **Study on dried pomegranate arils as influenced by cultivars, harvest maturity and storage of whole fruit**

- Physicochemical attributes, phytochemical properties and antioxidant capacity of dried pomegranate (*Punica granatum*) arils as affected by cultivar (Paper 1)
  - Investigating the effects of harvest maturity stages on the quality of dried pomegranate aril (cv. Wonderful) (Paper 2)
  - Effects of cold storage of raw material on physicochemical, phenolic content and antioxidant capacity of hot-air and freeze-dried pomegranate arils (Paper 3)
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## PAPER 1

# Physicochemical attributes, phytochemical properties and antioxidant capacity of dried pomegranate (*Punica granatum*) arils as affected by cultivar

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### ABSTRACT

A comparative study of the physicochemical attributes, phytochemical properties and antioxidant capacity of dried arils from three pomegranate cultivars (Acco, Herskawitz and Wonderful) was conducted. The hot-air drying experiment was carried out at 60 °C, 19.6 % relative humidity and at air velocity of 1.0 m s<sup>-1</sup>. Dried pomegranate arils of each cultivar were assessed for colour, total soluble solids (TSS), titratable acidity (TA), pH, total phenolic content (TPC) and total anthocyanin content (TAC). The antioxidant capacity of dried arils was evaluated in radical scavenging activity (RSA) and ferric reducing ability power (FRAP) assays. The results showed that desirable quality attributes and functional properties of the investigated dried pomegranate arils were cultivar dependent. The TSS, TA and pH were in the range of 16.3 – 21.0 °Brix, 1.23 – 1.50 (% citric acid) and 3.36 - 3.85, respectively. ‘Wonderful’ had the highest TPC (113.0 mg GAE/ g), which was 7.4% and 10.4% higher than ‘Acco’ and ‘Herskawitz’, respectively. However, the TAC was not significantly ( $p > 0.05$ ) different amongst the cultivars (‘Wonderful’; 23.9 mg C3gE/g, ‘Herskawitz’; 20.8 mg C3gE/g, and ‘Acco’; 20.1 mg C3gE/g). All investigated cultivars had high antioxidant capacity; dried arils from ‘Acco’ had 28% more RSA than the other cultivars, while ‘Wonderful’ and ‘Herskawitz’ had the highest FRAP.

Keywords: Cultivar, dried arils, total soluble solids, colour, anthocyanin

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### 1. Introduction

Pomegranate (*Punica granatum*) belongs to the plant family Punicaceae and are widely spread over the geographical location of the northern hemisphere (Fawole et al., 2012). The fruit, because of its high nutritional and functional values such as high sugar content, proteins, organic acids, polysaccharides, minerals, vitamin C and polyphenols, has gained a remarkable acceptance from consumers. (Lansky and Newman, 2007; Opara et al., 2009; Miguel et al., 2010; Fawole and Opara, 2013). Pomegranate arils are eaten fresh and used in various kinds of food, including jams, jellies, wine, and beverages (Aarabi et al., 2008; Opara et al., 2009; Mousavinejad et al., 2009).

The consumption of pomegranate arils in a higher amount has been associated with improved human health due to its active phenolic compounds such as antioxidant, anti-inflammatory, anti-carcinogenic and anti-hypertension properties (Fawole et al., 2012; Gil et al., 2000; Kaur et al., 2006; Duman et al., 2009; Viuda-Martos et al., 2010).

As a result of the short postharvest life (5 to 7 d) of pomegranate arils (Caleb et al., 2012), there has been an increasing focus on drying of pomegranate for extended shelf-life. This has been driven by a shift in consumer preference for minimally processed food products, especially fruit-made snacks (Wojdylo et al., 2016). The quality of dried pomegranate arils is highly dependent on the final moisture content of the products. Like in many other dried products, low moisture content does not only prevent the development of micro-organisms, but it also reduces the weight and volume of the dried product during transportation and storage. In Iran and India, pomegranate arils are dried into a nutrient-dense snack, called Anardana and are available mainly at health and food stores (Sharma and Thakur, 2016). Also, it is essential to achieving a characteristic flavour of the dried product (Sabarez et al., 2000).

In the food industry, the cultivars utilised to produce confectioneries are often characterised by several factors including large fruit size, high sugar content, intense colour, and crispness as well as the high phytochemical and antioxidant attributes (Gobbi et al., 1996; Newman et al., 1996; Crivelli et al., 1998). For instance, the critical feature used for the selection of cultivar for drying among the fruits of the Rosaceae family is the sorbitol content (Forni et al., 1992). A study by Wilford et al. (1997) on the changes in glucose, fructose, sucrose, and sorbitol during dehydration of d'Agen prunes reported an increasing proportional amount in both hydrolysis products (fructose and glucose). Three varieties of jujube fruit identified as GAL, MSI, and PSI were studied by Wojdylo et al. (2016), and the authors concluded that the best varieties were PSI and GAL based on the biochemical component and sensory quality, respectively. Similarly, the effect on the quality characteristics of different prune cultivars was studied by Cinquanta et al. (2002), and the authors noted that Stanley prunes had the best characteristic feature for drying because of its total sugar and sorbitol content. At the same time, the Angelino plums resulted in the highest total phenolic and anthocyanin content. Furthermore, according to Mahayothee et al. (2007), the quality of sulphite-free dried mango slices was greatly influenced by variety, ripening condition and



ripening stage of Mangoes ‘Kaew’, and the cultivar was more appropriate to undergo dehydration better than ‘Nam Dokmai’ due to superior trans- $\beta$ -carotene contents of the products.

Due to the inherent genetic differences that surround pomegranate cultivars as well as climate effect on the phenolic content and antioxidant capacity of the fruit (Al-Said et al., 2009; Holland et al., 2009; Opara et al., 2009), scientific evaluation of the commercially grown cultivar is a necessity to select and market fruit suitable for dried aril processing. Therefore, this study aimed to investigate the physicochemical attributes, phytochemical properties and antioxidant capacity of three commercially grown pomegranate cultivars in South Africa. The specific objectives were to assess the sensorial attributes including colour, chemical (soluble solids contents, pH and acidity), as well as the phenolic content and the antioxidant capacity of dried arils.

## **2. Materials and Methods**

### **2.1. Fruit material and processing**

Three pomegranate cultivars (Acco, Herskawitz and Wonderful), classified as sweet, sour and sweet-sour, respectively, were harvested at commercial harvest ( $>13^\circ\text{Brix}$ ) between February and April 2018, from Ms Elrita Venter’s orchard, Koringberg, South Africa ( $33^\circ02'00''$  S,  $18^\circ40'00''$  E). The fruit were sorted for uniformity in size, shape and colour and transported in an air-conditioned vehicle to the Postharvest Technology Laboratory at Stellenbosch University. Pomegranate arils were manually extracted and used for immediate processing. Arils from three cartons of fruit (replicates), each containing 10 to 12 fruits, were dried per cultivar. The quality indices of the pomegranate fruit cultivars and moisture content of the investigated cultivars are presented in Table 1.

Aril moisture content was measured in triplicates by drying samples (100 g) per cultivar in an oven (Model nr. 072160, Prolab Instruments, Sep Sci., South Africa) at  $60^\circ\text{C}$ , 19.6 % relative humidity and  $1.0\text{ m s}^{-1}$  constant air velocity. Drying was stopped at the recommended moisture content (between 10 and 12 %) (Kingsly et al., 2006).

### **2.2. Extraction of samples**

Dried pomegranate arils were ground into powder using liquid nitrogen followed by extraction of 5 g sample in 50 mL distilled water. The mixture was vortexed for 5 min, sonicated

for 15 min in an ultrasonic bath before centrifugation at 10 000 rpm and 4 °C for 25 min to recover the supernatant for TSS, TA and pH measurements. For the phytochemical properties and antioxidant capacity, the same extraction procedure was followed using 50 % methanol.

### 2.3. Determination of total soluble solids (TSS), titratable acidity (TA) and pH

TSS was measured using a digital hand refractometer (model PT-32; ATAGO, Tokyo, Japan) with the range of 0-32 °Brix. TA was measured by titrating the extract against 0.2 N of sodium hydroxide (NaOH) to a pH of 8.2 using a Metrohm 862 Compact titrosampler (Herisau, Switzerland). The pH value was measured using a pH meter (Crison, Barcelona, Spain).

### 2.4. Colour measurement

The colour of the dried arils was determined using a chromo-meter (Minolta model CR-200, Osaka, Japan) to obtain the colour attributes;  $L^*$  (brightness/darkness),  $a^*$  (redness/greenness) and  $b^*$  (yellowness/blueness). The colour parameters chroma  $C^*$  and hue angle  $h^\circ$  were calculated using equations 1 and 2 (Fawole and Opara, 2013; Pathare et al., 2013), respectively:

$$C^* = (a^{*2} + b^{*2})^{1/2} \quad (1)$$

$$h^\circ = \tan^{-1} (b^* / a^*) \quad (2)$$

The results were expressed as means  $\pm$  S.E. of determinations obtained.

### 2.5. Phytochemical analysis

#### 2.5.1. Determination of total phenolic content (TPC)

TPC of arils was determined by the Folin–Ciocalteu method using a methanolic extract of dried arils. The supernatant (0.05 mL) was mixed with 0.45 mL of 50 % methanol in a test tube followed by the addition of 0.5 mL Folin–Ciocalteu after 2 min. The mixture was then vortexed and kept in the dark for 10 min before adding 2 %  $\text{Na}_2\text{CO}_3$  and further incubation for 40 min in the dark. The absorbance of each sample was read at 520 nm in a UV-visible spectrophotometer (Thermo Scientific technologies, Madison, USA) against a blank containing 50 % methanol. Absorbance was compared with a standard curve (Gallic acid, 0 - 10 mg), and the results were expressed as mg gallic acid equivalent per gram pomegranate dry matter (mg GAE/g DM) (Fawole et al., 2012).

### 2.5.2. Determination of total anthocyanin content

Total anthocyanin content (TAC) was quantified using the pH differential method (Wrolstad, 1993). In triplicates, extract (1 mL) was mixed with 9 mL of pH 1.0 and pH 4.5 buffers separately. Absorbance was measured at 520 and 700 nm in pH 1.0 and 4.5 buffers and the result was expressed as cyanidin 3-glucoside using equation 3.

$$A = (A_{510} - A_{700})_{pH\ 1.0} - (A_{510} - A_{700})_{pH\ 4} \quad (3)$$

$$\text{Total monomeric anthocyanin (mg/mL)} = \frac{A \times MW \times DF}{\epsilon \times L}$$

where A=Absorbance,  $\epsilon$ =Cyd-3-glucoside molar absorbance (26,900), MW=anthocyanin molecular weight (449.2), DF=dilution factor, and L=cell path length (1 cm). The final results are expressed as equivalent per gram dry matter (mg C3gE/g DM).

## 2.6. Antioxidant capacity

### 2.6.1. Radical-scavenging activity (RSA)

The RSA was carried out in triplicate, according to Fawole et al. (2012). Briefly, under dim light, aqueous methanolic extract of dried aril (0.015 mL) was diluted with methanol (0.735 mL) in test tubes followed by the addition of methanolic DPPH solution (0.75 mL, 0.1 mM). The mixtures were incubated at room temperature for 30 min in the dark, and the absorbance was measured at 517 nm using a UV-vis spectrophotometer. Absorbance was compared with the standard curve (Trolox equivalent, 0 – 2.0 mM). The free-radical activity of dried aril was expressed as Trolox equivalent (mM) equivalent per gram dry matter (mM TE/g DM).

### 2.6.2. Ferric-ion reducing antioxidant power (FRAP)

The antioxidant power of dried aril was measured calorimetrically according to Benzie and Strain (1996) and Fawole et al. (2012). The FRAP working solution used contained freshly prepared mixtures of 300 mM acetate buffer (50 mL), 10 mM 2,4,6-tripyridyl-s-triazine (TPTZ) (5 mL) and 20 mM ferric chloride (5 mL). In triplicates, diluted aqueous methanolic dried aril extracts (0.15 mL) were added to 2.85 mL of the FRAP working solution before incubation in the dark for 30 min. The reduction of the  $\text{Fe}^{3+}$ -TPTZ complex to a coloured  $\text{Fe}^{2+}$ -TPTZ complex at low pH by dried aril extracts was monitored by measuring the absorbance at 593 nm. Trolox (0 - 10 mM) was used for the calibration curve, and the results were expressed as Trolox (mM) equivalents per gram dry matter (mM TE/g DM).

## 2.7. Statistical analysis

Data were processed with STATISTICA (Statistica 13.0, StatSoft Inc., Tulsa, OK, USA) and presented as means  $\pm$  standard error. All analysis was done in triplicates. The data were subjected to analysis of variance (ANOVA), and means were separated according to Fisher's LSD test at a level of significance of 95%. GraphPad Prism software 4.03 (GraphPad Software, Inc., San Diego, USA) was used for graphical presentations.

## 3. Results and discussion

### 3.1. Physicochemical properties of fresh pomegranate aril

Table 1 shows the major physicochemical attributes of the investigated cultivars. 'Wonderful' had the highest moisture content (78.51), followed by 'Acco' (56.65) and 'Herskawitz' (54.86). The titratable acidity (TA) was in the order of 'Herskawitz'; 1.54 > 'Wonderful'; 1.42 > 'Acco'; 1.36. However, 'Acco' had the highest total soluble solid (16.2) compared to 'Herskawitz' (14.8) and 'Wonderful' (15.8). Similarly, Fawole et al. (2014) reported variations in the TSS, TA and other physicochemical attributes of eight cultivars of pomegranate arils. The weight of arils was not statistically different amongst the investigated cultivars. Both 'Herskawitz' and 'Wonderful' had higher lightness ( $L^*$ ) in comparison with 'Acco' while the redness of fresh pomegranate arils was not statistically different amongst the cultivars. Overall, the parameters reflected distinct genetic differences amongst the cultivars.

### 3.2. Aril moisture changes with drying time

Figure 1 shows the experimental drying behaviour of the three cultivars ('Acco', 'Herskawitz' and 'Wonderful'). Dried aril cultivars showed the different drying behaviour along drying time. At the end of the drying process, 'Wonderful', which had the highest moisture content, showed the least drying time (11 h) to reach 10 % moisture, while 'Acco' and 'Herskawitz' had the most extended drying times (15 and 20 h), respectively. Variation in the drying time could be due to the strength of the aril sac. Moisture change over time is described by two major factors which are transport properties of the material and the drying air (Guiné et al., 2007). Moreover, the initial structure of the arils belonging to each cultivar could not be maintained, as drying ruptured the structure of pomegranate arils at different times. Similar results were reported by

(Cruz et al., 2015), who observed a variation in the drying times for ‘Golden Delicious’ and ‘Granny Smith apples’ at 60 °C.

### 3.3. Total soluble solids, titratable acidity and pH

In general, dried aril characteristics of the pomegranate cultivars investigated were significantly ( $p < 0.05$ ) different (Table 2). The total soluble solids content ranged from 16.3 to 21.0°Brix, the highest being ‘Wonderful’ and the least being ‘Herskawitz’. Total soluble solids (TSS) in dried pomegranate arils increased for all cultivars after drying. The variation in TSS values after drying could be due to the difference in the concentration of soluble solids within the arils. During drying, sucrose is thermally degraded and hydrolysed to glucose and fructose (Crivelli et al., 1998), increasing sweetness of the dried product. It is thus logical to assume that sweetness of the dried pomegranate arils will vary depending on the fruit cultivars. This observation is in agreement with Crivelli et al. (1998) who reported differences in sweetening behaviour among plum varieties during drying and attributed the observation to the co-occurrence of thermal degradation and hydrolysis of sucrose to glucose and fructose. A similar observation was also reported by Cinquanta et al. (2002), who attributed an increase in sweetness in plums after drying. The TSS values obtained in this study were higher than those reported (14.4 – 15.1°Brix) for dried pomegranate arils grown in India (Poyrazoglu et al., 2002).

The highest titratable acidity was found in ‘Herskawitz’ (1.50 % citric acid), while ‘Acco’ contained the lowest amount (1.23 % citric acid) (Table 2). Also, a general decrease in titratable acidity was observed in dried pomegranate arils as compared to fresh arils. For instance, in Acco cultivar, titratable acidity decreased from 1.36 to 1.23 % citric acid, Herskawitz; 1.54 to 1.50 % citric acid, and Wonderful; 1.42 to 1.40 % citric acid. The observed decrease in acidity could be attributed to the degradation of organic acids during drying, a phenomenon in agreement with (Kumar et al., 2009) who reported a decrease in the acidity of osmo-vacuum dehydrated anola. The authors attributed the decrease to the conversion of citric acid to acetic acids. The acidity levels observed in the dried arils were accompanied by relatively high pH values which ranged from 3.36 to 3.85 (Table 2).

Similarly, the dynamics of the TSS and TA reflected on their ratios. ‘Acco’ and ‘Wonderful’ had higher sugar to acid ratios compared to ‘Herskawitz’. Low TSS/TA in ‘Herskawitz’ could be due to the slow rate of acid degradation. Study on sulphite-free dried mango

slices by (Mahayothee et al., 2007) revealed that a decrease in the sugar to acid ratio was as a result of acid degradation.

### 3.4. Colour attributes

The colour attributes of the investigated pomegranate cultivars are shown in Table 3. Dried aril colour did not vary significantly ( $p < 0.05$ ) in the colour parameters  $L^*$ ,  $a^*$  and  $C^*$  among the pomegranate cultivars except in  $h^\circ$ . This was expected as the observed colour variation was also not observed visually. Colour is an important attribute used for assessing acceptability, marketability and consumer preference in pomegranate (Opara et al., 2009, Fawole and Opara, 2013; Pathare et al., 2013). In this study, the lightness ( $L^*$ ) of dried arils for ‘Wonderful’ was the highest among the cultivars reported. The CIE  $a^*$  (+) value, which indicates the redness of the dried arils, ranged between 13.5 and 18.4 (Table 3). This is an indication of a change in aril colour from light red to dark red. ‘Wonderful’ had the highest  $a^*$  value while ‘Acco’ had the least. Furthermore, the values of aril colour intensity ( $C^*$ ) varied markedly among the cultivars; the highest and the least colour intensity were found in ‘Wonderful’ and ‘Acco’, respectively. Colour purity ( $h^\circ$ ) was highest for ‘Acco’ while the lowest was measured for ‘Wonderful’. The observed changes in aril colour parameters could be attributed to prolonged drying, and consequently, imparting on the colour change (Maillard reactions) during drying (Cinquanta et al., 2002; Sharma et al., 2013).

### 3.5. Phytochemical analysis

#### 3.5.1. Total phenolic content (TPC)

Pomegranate fruit is one of the richest sources of polyphenols. Several researchers have reported considerable varying amounts of polyphenols in the fruit (Fawole and Opara, 2013; Mousavinejad et al., 2009; Gil et al., 2000; Poyrazoglu et al., 2002; Tezcan et al., 2009 and Tehranifar et al., 2010). In dried arils, the drying process influenced the final content of total phenolic content, causing significant ( $p < 0.05$ ) differences between the cultivars (Figure 2). The total phenolic content expressed as milligrams of gallic acid equivalent (GAE) per g dried arils ranged from 101.3 – 113.0 g, ‘Wonderful’ having 7.4 and 10.4 % of TPC concentration than ‘Acco’ and ‘Herskawitz’, respectively. The observed differences in TPC could also be attributed to the cultivar difference. According to Cinquanta et al. (2002), 29 % of the total phenolic content was reported in dried Angeleno plum pulps compared to cvs. Stanley and Empress.

### 3.5.2. Total anthocyanin content

The results in Figure 3 showed considerable amounts of total anthocyanin content (TAC) expressed as cyanidin 3- glucoside equivalents (mg C3gE/g) in the investigated pomegranate cultivars. The total anthocyanin content was higher in Wonderful cultivar (23.9 mg C3gE/g) compared to ‘Acco’ (20.1 mg C3gE/g) and ‘Herskawitz’ (20.8 mg C3gE/g). The presence of anthocyanin is an indication of red pigments in pomegranate fruit. The lower values of total anthocyanin content observed in ‘Acco’ and ‘Herskawitz’ could be due to the sensitivity to the heat applied on the arils. Degradation by the oxidative process, in which enzymatic and heat-related conditions are involved could result in the lower value of total anthocyanin content (Cinquanta et al., 2002) as observed in the studied cultivars.

### 3.6. Antioxidant capacity

The antioxidant capacity of pomegranate dried arils showed significant differences among the cultivars ( $p < 0.05$ ) as presented in Fig. 4a. The radical scavenging activity of polyphenolic dried arils ranged between 26.4 to 37.2 mM TE/g. ‘Acco’ had the higher RSA than ‘Herskawitz’ and ‘Wonderful’. In the FRAP activity, ‘Wonderful’ had the highest FRAP (4.32 mM TE/g) followed by ‘Herskawitz’ (4.24 mM TE/g) while ‘Acco’ had the least (3.10 mM TE/g). The FRAP activity in ‘Wonderful’ was approximately 28.2 and 1.9 % higher than in Acco and Herskawitz cultivars (Fig. 4b). The variations observed in the antioxidant capacity could be attributed to differences in reaction kinetics, the steady state antioxidant capacity as well as the reductive substrates (Ozgen, et al., 2008). The high antioxidant capacity exhibited by ‘Wonderful’ could be due to non-enzymatic browning reactions (Maillard), often associated with compounds formation with a strong antioxidant capacity (Manzocco et al., 2000). In dried pomegranate arils, cultivar having the lowest TAC resulted in a corresponding decrease in the antioxidant capacity exhibited by FRAP activity. Similar to the study by Du et al. (2013) reported that the cultivar with the least phenolic content resulted in the least antioxidant capacity RSA and FRAP. In addition, the occurrence of oxidative decomposition through Maillard reaction or thermal degradation, as a result of drying, has been reported to have a detrimental effect on the bioactive components of dried fruit (Al-Farsi et al., 2005).

#### 4. Conclusion

This study has demonstrated that the cultivars investigated were characterised by distinct qualities related to colour, chemical and phytochemical properties. Based on the retention of TSS, TPC, TAC and FRAP activity, Wonderful cultivar performed better than ‘Herskowitz’ and ‘Acco’. Additionally, the investigated bioactive properties reported in this study produce the first documentation and scientific evidence which could be used to define the phenolic content and antioxidant profiling of dried arils obtained from pomegranate cultivars grown in South Africa. This result could assist in the development of scientific procedures for processing and quality assessment of dried pomegranate arils.

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**Table 1**

Quality indices of fresh pomegranate arils for different cultivars at commercial maturity

Attribute	Acco	Herskawitz	Wonderful
Moisture (%)	56.65 ± 1.22 <sup>b</sup>	54.86 ± 1.59 <sup>b</sup>	78.51 ± 1.03 <sup>a</sup>
pH	3.01 ± 0.05 <sup>a</sup>	3.11 ± 0.08 <sup>a</sup>	3.06 ± 0.06 <sup>a</sup>
TA (% citric acid)	1.36 ± 0.01 <sup>c</sup>	1.54 ± 0.01 <sup>a</sup>	1.42 ± 0.03 <sup>b</sup>
TSS (°Brix)	16.2 ± 0.09 <sup>a</sup>	14.8 ± 0.24 <sup>b</sup>	15.8 ± 0.15 <sup>a</sup>
TSS/TA	11.9 ± 0.06 <sup>a</sup>	9.64 ± 0.17 <sup>c</sup>	11.2 ± 0.19 <sup>b</sup>
Weight of aril (g)	0.33 ± 0.01 <sup>a</sup>	0.31 ± 0.01 <sup>a</sup>	0.39 ± 0.05 <sup>a</sup>
Aril Lightness (L*)	23.8 ± 0.74 <sup>b</sup>	29.7 ± 1.13 <sup>a</sup>	28.4 ± 1.71 <sup>a</sup>
Aril redness (a*)	15.1 ± 1.07 <sup>a</sup>	16.3 ± 0.79 <sup>a</sup>	13.9 ± 0.83 <sup>a</sup>
Chroma (C*)	18.5 ± 0.54 <sup>ab</sup>	19.9 ± 0.71 <sup>a</sup>	17.4 ± 0.44 <sup>b</sup>
Hue (h°)	32.2 ± 0.25 <sup>a</sup>	34.9 ± 1.21 <sup>a</sup>	37.6 ± 3.45 <sup>a</sup>

Titrateable acidity (TA), Total soluble solids (TSS). Data presented as means ± SE in each column followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.

**Table 2**

Chemical properties of dried pomegranate arils of different cultivars

Cultivar	TSS (°Brix)	TA (% citric acid)	TSS/TA	pH
Acco	19.3±0.88 <sup>ab</sup>	1.23±0.07 <sup>b</sup>	15.9±1.64 <sup>a</sup>	3.36±0.00 <sup>c</sup>
Herskawitz	16.3±0.88 <sup>b</sup>	1.50±0.06 <sup>a</sup>	10.9±0.59 <sup>b</sup>	3.85±0.01 <sup>a</sup>
Wonderful	21.0±1.00 <sup>a</sup>	1.40±0.01 <sup>ab</sup>	15.0±0.71 <sup>a</sup>	3.39±0.01 <sup>b</sup>

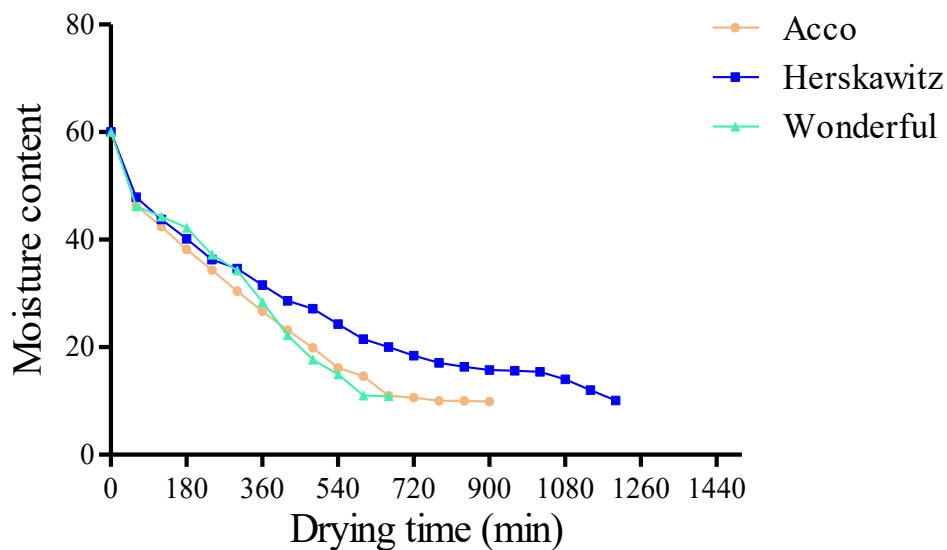
TSS; total soluble solids, TA; titrateable acidity. Data presented as means ± SE in each row followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.

**Table 3**

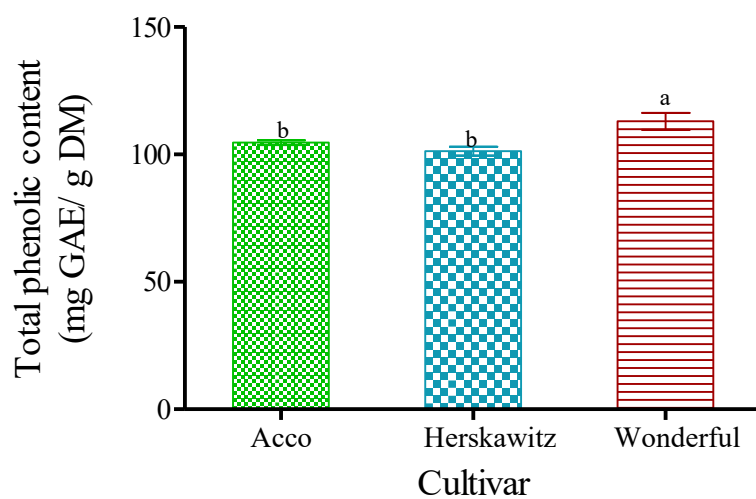
Colour attributes of dried pomegranate arils obtained from different cultivars

Cultivar	L*	a*	C*	h°
Acco	21.5±0.77 <sup>b</sup>	13.5±0.15 <sup>a</sup>	16.8±0.05 <sup>a</sup>	36.4±0.75 <sup>a</sup>
Herskawitz	25.8±2.25 <sup>ab</sup>	15.4±2.52 <sup>a</sup>	18.0±2.10 <sup>a</sup>	31.9±4.75 <sup>ab</sup>
Wonderful	28.1±1.86 <sup>a</sup>	18.4±0.49 <sup>a</sup>	20.0±0.68 <sup>a</sup>	23.2±1.10 <sup>b</sup>

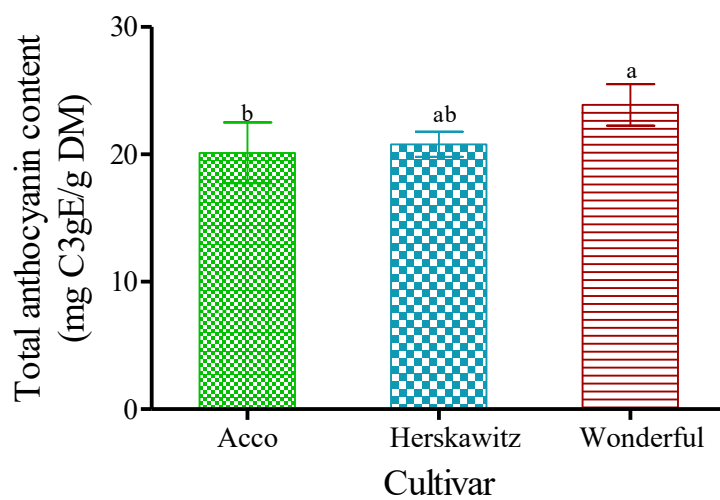
L\*: lightness, a\*: redness, C\*: chroma, h°: hue angle. Data presented as means ± SE in each row followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.



**Fig. 1.** Moisture content vs drying times (min) at 60 °C, 19.6 % relative humidity (RH) for ‘Acco’, ‘Herskawitz’ and ‘Wonderful’ pomegranate dried arils.

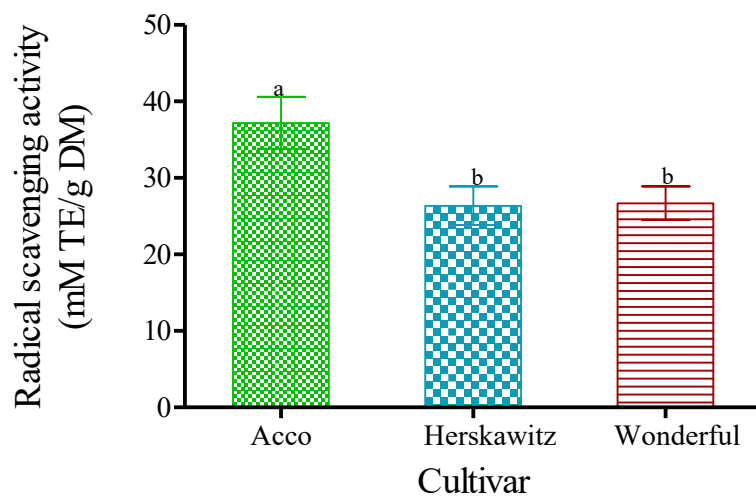


**Fig. 2.** Total phenolic content of dried pomegranate arils obtained from different cultivars. GAE, gallic acid equivalent, DM; dry matter (dried pomegranate arils).

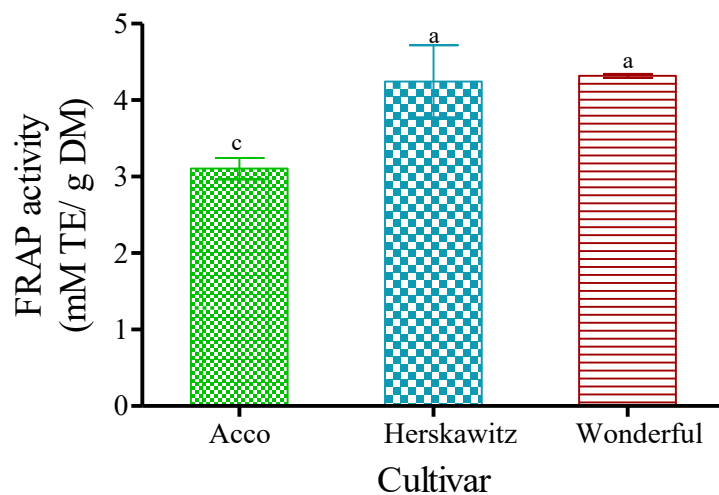


**Fig. 3.** Total anthocyanin content of dried pomegranate arils obtained from different cultivars. C3gE; cyaniding-3glucoside equivalent, DM; dry matter (dried pomegranate arils).

(a)



(b)



**Fig. 4.** Antioxidant capacity (a) RSA and (b) FRAP activity of dried pomegranate arils obtained from different cultivars. RSA; radical scavenging activity, FRAP; ferric reducing antioxidant power, TE; trolox equivalent, DM; dry matter (dried pomegranate arils).

## PAPER 2

### Effect of harvest maturity on the physicochemical properties, phenolic content and antioxidant capacity of dried pomegranate (*Punica granatum*) arils (cv. Wonderful)

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#### Abstract

This study investigated the quality attributes of dried pomegranate arils obtained at early, commercial and late harvest. The hot-air drying experiments were carried out at 60°C, 19.6% relative humidity and air velocity of 1.0 m s<sup>-1</sup>. Dried arils for each harvest maturity were assessed for colour, total soluble solids (TSS), titratable acidity (TA), pH, phenolics and antioxidant capacity. Results showed that major quality attributes in dried arils are dependent on harvest maturities. TSS, TA and pH were in the range of 14.8 – 22.2°Brix, 1.31 – 1.77 (% citric acid) and 3.33 - 3.65, respectively. Red colour (a\*) of dried aril was significantly higher ( $P < 0.05$ ) at commercial (19.4) and late harvest (19.3) than with early harvest (14.5). Also, the commercial harvest had the highest chroma value (22.3) for dried aril in comparison with other harvest maturities (early, 18.1; late, 21.9). Commercial harvest had the highest TPC with 18.6% and 8.7% higher than early and late harvest, respectively. Also, the early harvest had least TAC with commercial and late harvest having between 1.2 and 1.1-fold more than early harvest. Overall, the parameters reflected distinct differences in quality with harvest maturity.

Keywords: dried arils; harvest maturity; total soluble solids; anthocyanin; antioxidants

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#### 1. Introduction

Pomegranate (*Punica granatum*) fruit is popularly consumed as fresh arils; however, due to its short shelf-life (between 5 to 7d), there is an increasing interest in the drying of the arils for extended postharvest shelf-life. This has, in recent years, been driven by a shift in consumer's preference towards consumption of processed products from fruit, particularly fruit snacks (Wojdyło et al., 2014). Quality and shelf-life of dried pomegranate arils is highly dependent on the final moisture content of the products. Like in many other dried products, low moisture content does not only halt the activity of microorganisms but also result in lighter weight product during transportation and storage. In addition, drying also helps to achieve a characteristic flavour

(Sabarez et al., 2000). In Iran and India, pomegranate arils are dried into a nutrient-dense snack, called ‘Anardana’ and are available mainly at health and food stores (Sharma and Thakur, 2016). Studies have been conducted to optimize drying parameters such as temperature and time, as well as pretreatments for pomegranate arils (Thakur et al., 2010). However, limited studies have reported the impact of fruit maturity on the quality of dried products (Mahayothee et al., 2007).

Non-climacteric fruits, such as pomegranate, are not characterized by a continued ripening process after harvest. In addition to the effect of agronomic practices such as harvest dates, cultivar, season and climatic condition, postharvest factors including storage temperatures and duration as well as packaging materials also play a significant role in influencing the postharvest quality of pomegranate fruit (Mirdehghan and Rahemi, 2007). Thus, several quality characteristics determine consumer’s and processor’s preferences in the acceptability of pomegranate fruit. These qualities include chemical constituents such as sugar concentration, acidity and flavour, and physical appearance attributes such as colour and size (Al-Said et al., 2009). Although on-farm sorting and grading by colour and size is applied in the pomegranate fruit industry, the most crucial index for quality assessment to meet the market requirement are the internal attributes which include aril colour, total soluble solids content and titratable acidity (Fawole and Opara, 2013). For instance, in Ruby cultivar, juice content and total soluble solids increased with prolonged harvest dates, with concomitant decreases in titratable acidity and total phenolic concentrations (Fawole and Opara, 2013a). Furthermore, the concentrations of the phytochemical properties have been of interest due to their effect as health-giving properties (Fawole and Opara, 2013b). For instance, variations in the content of total phenolic and total anthocyanin contents as well as the antioxidant capacity were observed among the maturity status of pomegranate cultivar Bhagwa (Fawole and Opara, 2013b).

Due to the challenges mostly associated with the chemical properties of fruit during drying, for instance, fruit harvested at late maturity with high moisture and sugar content requires more energy during drying. This could lead to browning (sugar caramelization) and cause damage to the physicochemical properties of the final product. Hence, there is a need to investigate the maturity stages for dried aril processing, in consideration of quality and energy requirement. Therefore, this study aimed to evaluate the physicochemical attributes, phytochemical properties and antioxidant capacity of dried pomegranate aril ‘Wonderful’ at three harvest maturity stages. The specific



objectives were to evaluate the sensorial attributes including colour, chemical (total soluble solids, pH and titratable acidity), phenolic contents and the antioxidant capacity of dried arils to assess the health-giving properties of the dried product. This information is essential in the agro-processing industry to improve the flavour characteristics of pomegranate product.

## **2. Materials and Methods**

### **2.1. Fruit material and processing**

Pomegranate cv. Wonderful was harvested at three different maturities which are 2 weeks before commercial harvest which is regarded as early harvest (H1), at commercial harvest (mid-harvest; H2) and 2 weeks after commercial harvest (late harvest maturity; H3) between January and April 2019, from Blydeverwacht orchard, Wellington, South Africa (33°01'00" S, 18°58'59" E). Fruit were sorted for uniformity in size, shape and colour and transported in an air-conditioned vehicle to the Postharvest Technology Laboratory at Stellenbosch University. Pomegranate arils were manually extracted from the fruit and used for immediate processing. Arils from three cartons of fruit (replicates), each containing 10 to 12 fruits, were dried per harvest maturity. The quality indices of pomegranate fruit at different harvest maturities are presented in Table 1. Aril moisture content was measured in triplicates by drying samples (100 g) per harvest maturity in an oven (Model nr. 072160, Prolab Instruments, Sep Sci., South Africa) operating at 60°C, 19.6% relative humidity and  $1.0 \text{ m s}^{-1}$  constant air velocity. Drying was stopped between 10 and 12% recommended moisture content (Kingsly et al., 2006).

### **2.2. Extraction of samples**

Dried pomegranate arils were ground into powder using liquid nitrogen followed by extraction of 5 g sample in 50 mL distilled water. The mixture was vortexed for 5 min and sonicated for 15 min in an ultrasonic bath. This was followed by centrifugation at 10 000 rpm for 25 min and recovery of the supernatant for TSS, TA and pH measurements. For phytochemical and antioxidant capacity, the same extraction procedure was followed using 50% methanol.

#### **2.2.1. Determination of total soluble solids (TSS), titratable acidity (TA) and pH**

TSS was measured using a digital hand refractometer (model PT-32; ATAGO, Tokyo, Japan) with the range of 0-32°Brix which was blanked with distilled water. For TA, two millilitres of the supernatant was diluted in 70 mL of distilled water and titrated against 0.2 N of sodium

hydroxide (NaOH) to a pH of 8.2 using a Metrohm 862 Compact titrosampler (Herisau, Switzerland). BrimA index, a variant of TSS/TA and a criterion for acceptance of fruit juice, which is expressed as  $\text{BrimA} = \text{TSS} - k * \text{TA}$ , where  $k$  is the tongue's sensitivity index normally ranging from 2 - 10 (Fawole and Opara, 2012). In this study, a  $k$  value of 2 was used to avoid negative BrimA index. The pH value of dried pomegranate aril was measured using a pH meter (Crison, Barcelona, Spain).

### 2.2.2. Colour measurement

Colour of the dried was determined on arils placed in a colourless Petri dish. Measurement was carried out using a chromo-meter (Minolta model CR-200, Osaka, Japan) to obtain the colour attributes;  $L^*$  (brightness/darkness),  $a^*$  (redness/greenness) and  $b^*$  (yellowness/blueness). The colour parameters chroma  $C^*$  and hue angle  $h^\circ$  were calculated using equations 1 and 2 (Fawole and Opara, 2013a; Pathare et al., 2013).

$$C^* = (a^{*2} + b^{*2})^{1/2} \quad (1)$$

$$h^\circ = \tan^{-1} (b^* / a^*) \quad (2)$$

Results were expressed as means  $\pm$  S.E. of determinations obtained.

## 2.3. Phytochemical analysis

### 2.3.1. Determination of total phenolic content (TPC)

TPC of arils was determined by the Folin–Ciocalteu method using a methanolic extract of dried arils. The supernatant (0.05 mL) was mixed with 0.45 mL of 50% methanol in a test tube followed by the addition of 0.5 mL Folin–Ciocalteu after 2 min. The mixture was then vortexed and kept in the dark for 10 min before adding 2 %  $\text{Na}_2\text{CO}_3$  and further incubation for 40 min in the dark. The absorbance of each sample was read at 520 nm in a UV-visible spectrophotometer (Thermo Scientific technologies, Madison, USA) against a blank containing 50% methanol. Absorbance was compared with a standard curve (Gallic acid, 0 - 10 mg), and results were expressed as mg gallic acid equivalent per gram pomegranate dry matter (mg GAE/g DM) (Fawole et al., 2012).

### 2.3.2. Determination of total anthocyanin content

Total anthocyanin content (TAC) was quantified using the pH differential method (Wrolstad, 1993). In triplicates, extract (1 mL) was mixed with 9 mL of pH 1.0 and pH 4.5 buffers separately. Absorbance was measured at 520 and 700 nm in pH 1.0 and 4.5 buffers and the result was expressed as cyanidin 3-glucoside using equation 3.

$$A = (A_{510} - A_{700})_{pH\ 1.0} - (A_{510} - A_{700})_{pH\ 4} \quad (3)$$

$$\text{Total monomeric anthocyanin (mg/mL)} = \frac{A \times MW \times DF}{\epsilon \times L}$$

where A=Absorbance,  $\epsilon$ =Cyd-3-glucoside molar absorbance (26,900), MW=anthocyanin molecular weight (449.2), DF=dilution factor, and L=cell path length (1 cm). Final results are expressed as equivalent per gram dry matter (mg C3gE/g DM).

## 2.4. Antioxidant capacity

### 2.4.1. Radical scavenging activity (RSA)

The RSA was carried out in triplicate, according to Fawole and Opara (2012). Briefly, under dim light, aqueous methanolic extract of dried aril (0.015 mL) was diluted with methanol (0.735 mL) in test tubes followed by the addition of methanolic DPPH solution (0.75 mL, 0.1 mM). The mixtures were incubated at room temperature for 30 min in the dark, and the absorbance was measured at 517 nm using a UV-vis spectrophotometer. Absorbance was compared with the standard curve (Trolox equivalent, 0 – 2.0 mM). The free-radical activity of dried aril was expressed as Trolox equivalent (mM) equivalent per gram dry matter (mM TE/g DM).

### 2.4.2. Ferric-ion reducing antioxidant power (FRAP)

The antioxidant power of dried aril was measured calorimetrically according to Benzie and Strain (1996); Fawole and Opara (2012). The FRAP working solution contained mixtures of 300 mM acetate buffer (50 mL), 10 mM 2,4,6-tripyridyl-s-triazine (TPTZ) (5 mL) and 20 mM ferric chloride (5 mL) was freshly prepared and incubated in a water bath at 37°C before being used. In triplicates, diluted aqueous methanolic dried aril extracts (0.15 mL) were added to 2.85 mL of the FRAP working solution before incubation in the dark for 30 min. The reduction of the  $\text{Fe}^{3+}$ -TPTZ complex to a coloured  $\text{Fe}^{2+}$ -TPTZ complex at low pH by dried aril extracts was monitored by

measuring the absorbance at 593 nm. Trolox (0 - 10 mM) was used for the calibration curve, and the results were expressed as Trolox (mM) equivalents per gram dry matter (mM TE/g DM).

## 2.7. Statistical analysis

Data were processed with STATISTICA (Statistica 13.0, StatSoft Inc., Tulsa, OK, USA) and presented as means  $\pm$  standard error. All analysis was done in triplicates. Data was subjected to analysis of variance (ANOVA) and means were separated according to Fisher's LSD test at a level of significance of 95%. GraphPad Prism software 4.03 (GraphPad Software, Inc., San Diego, USA) was used for graphical presentations. The relationship among the measured fruit parameters were determined by subjecting data to Pearson's correlation test and principal component analysis (PCA) using XLSTAT software version 2012.04.1 (Addinsoft, France).

## 3. Results and discussion

### 3.1. Quality indices of fresh pomegranate fruit for early, commercial and late harvest maturities

The moisture content of early (H1), commercial (H2) and late (H3) pomegranate arils were 76.99%, 78.51% and 78.63% as shown in Table 1. The moisture content did not differ ( $p < 0.05$ ) significantly for all the harvest maturities. Pomegranate fruit at early harvest had the highest titratable acidity (1.62% citric acid), followed by commercial harvest (1.42% citric acid) and late harvest 1.37% citric acid. The trend showed a decrease in titratable acidity with prolong harvest maturity. Study by Fawole and Opara (2013a) reported that decrease in TA often results in the reduction of acid biosynthesis during fruit maturation and could result in increased sweetness highly desirable to pomegranate arils. However, the total soluble solids (TSS) was in the order of late (H3); 16.2 °Brix > commercial (H2); 15.8 °Brix > early (H3); 13.2 °Brix with no significant ( $p < 0.05$ ) differences between the harvest maturities (Table 1). This reflected the hypothesis that a decline in titratable acidity during fruit maturation increases the level of sweetness in the fruit. Increase in the TSS with increasing maturation is presented in simple term by a phenomenon related to starch synthesis and sugar hydrolysis (Kulkarni and Aradhya, 2005; Mphahlele et al., 2014). Similar results were reported by Zarei et al. (2011) that total soluble solids (TSS), often made of sugar increased significantly during three major fruit developmental stages in pomegranate 'Rabbab-e-Fars'. Fruit weight values ranged between 523.6 g and 548.6 g (Table 1). However, the weight of aril was not statistically ( $p < 0.05$ ) different amongst the harvest maturities.

Pomegranate aril lightness ( $L^*$ ) values were 27.6, 25.4 and 25.2 for early (H1), commercial (H2) and late (H3), respectively. The highest aril redness ( $a^*$ ; 18.1) was found at commercial harvest maturity and was not statistically different from late harvest maturity (Santra and Jain, 2012; Sharma and Thakur, 2016), while early harvest maturity had the least ( $a^*$ ; 26.5) (Table 1). The degree of colour changes observed at different harvest times in fruit could be due to the varying ratios of anthocyanin (Zhang, 2009). Overall, the parameters reflected distinct differences in quality with harvest maturity.

### **3.2. Aril moisture changes with drying time**

Figure 1 shows the drying curves of pomegranate arils at different harvest times (early; H1, commercial; H2 and late; H3). Moisture content decreases with drying time. Pomegranate arils at early maturities reached the desired 10 to 12% moisture content faster (600 min) compared to both commercial and late maturities (660 min) (Fig. 1). A faster drying rate attained by pomegranate arils at early harvest could be attributed to the low moisture content observed at fresh fruit (Table 1) compared to commercial and late maturities. The result from this study was comparable to (Santra and Jain, 2012) who reported about 7.5 to 14 h (450 to 840 min) drying time for arils dried between 55°C and 60°C, respectively.

### **3.3. Total soluble solids, Titratable acidity and pH**

The chemical attributes of dried arils at different harvest maturities are presented in (Table 2). Dried pomegranate arils at commercial (H2; 20.3 °Brix) and late harvest (H3; 22.2 °Brix) had significantly higher total soluble solids (TSS) than early harvest (H1; 14.8 °Brix) (Table 2). Given that TSS increases and TA decreases with fruit maturity, dried arils obtained from each of the maturity stages mirrored the TSS and TA contents of fruit. A similar result was also reported by Fageria et al. (2003) in their study on the effect of harvest maturity on sun-dried ker and attributed the increase in TSS with the advancement in fruit development to the accumulation of soluble carbohydrates and hydrolysis (conversion of starch into soluble sugars). Increase in TSS concentration with advancing fruit maturity is explained by a mechanism related to starch synthesis and sugar hydrolysis as fruit advances in maturation (Kulkarni and Aradhya 2005; Fawole and Opara, 2013a; Mphahlele et al., 2014). It is logical to obtain high sugars from commercial and late harvest with higher TSS. However, values of TSS reported in many studies were higher compared

to that of the present study. For instance, Dadarao et al. (2010) reported 32.16 to 36.98 °Brix. Furthermore, Santra and Jain (2012) reported 30.2 to 38.73 °Brix for pomegranate dried at 45°C to 60°C. This could be dependent on genetic variability, geographical location and also the moisture content of the finished product. Titratable acidity (TA) of commercial (1.31) and late harvest (1.42) were significantly lower compared to the early harvest maturity (1.77). The lower value of TA at both commercial and late harvest could be attributed primarily to fruit maturity. A gradual decrease in the TA of pomegranate fruit was also observed according to Fawole and Opara (2013a). The authors attributed the decrease in TA to fruit development and ripening. TSS/TA value is a crucial criterion that is also used for quality evaluation of pomegranate arils in the processing industry for formulation/fortification of food and beverage products (Al-said et al., 2009; Fawole and Opara, 2013a).

There was also a significant ( $p < 0.05$ ) difference in the TSS/TA among harvest maturities. As a result of changes in TSS and TA contents, the ratio of TSS/TA increased considerably in both commercial and late harvest maturities in comparison with an early harvest. Further, the increase between the last two maturity stages (commercial to late) was not significant. The criterion for product acceptability which is expressed as BrimA was significantly ( $p < 0.05$ ) different amongst fruit harvested at different maturities (Table 2). Both commercial and late maturity had higher BrimA (17.7 and 19.4), respectively, compared to the fruit at early harvest maturity (11.2). Also, in the pH of dried pomegranate arils, the same pattern as observed in the titratable acidity (TA) was observed (Table 2). Early maturity dried arils had a significantly higher amount of pH (3.65) than commercial (3.37) and late maturities (3.33). This could confirm the high acidity level during early harvest maturity compared to other harvest maturities.

### **3.4. Colour attributes**

Table 3 summarises the results of dried pomegranate aril at different harvest maturities. The values of lightness ( $L^*$ ) of dried pomegranate aril showed no significant differences amongst harvest maturities and  $L^*$  decreased with prolong harvest time. The degree of redness ( $a^*$ ) was significantly ( $p < 0.05$ ) higher for dried arils at commercial (19.4) and late harvest (19.3) than with early harvest maturity (14.5). A similar trend was also observed for chroma ( $C^*$ ) of dried arils. Commercial harvest had the highest  $C^*$  value of 22.3 in comparison with other harvest maturities (early; 18.1 and late; 21.9). This suggests that the intensity of red pigmentation increased significantly with prolonging harvest time which could be a good indicator for consumer's

acceptance of dried arils. A similar result was reported by Ledbetter (2011) who observed a significant decrease in the chroma value for dried apricot of medium maturity than either the least or most mature fruit classes. On the other hand, the hue ( $h^\circ$ ) was higher at early harvest compared to commercial and late harvest maturities (Table 3). This could be due to colour change during drying due to Maillard reactions (Ashebir et al., 2009).

### **3.5. Total phenolic content (TPC) and total anthocyanin content (TAC)**

Results showed that total phenolic content was significantly ( $p < 0.05$ ) different amongst harvest maturities of dried pomegranate arils (Fig. 2). Dried aril at commercial harvest had higher TPC (H2; 124.7 mg GAE/ g) than at early (H1; 101.5 mg GAE/ g) and late (H3; 113.9 mg GAE/ g) harvest maturities. However, dried arils at commercial harvest were not statistically different from late harvest maturity. An increase in TPC with maturity could be due to the reduction in the astringency of arils, which is a desirable sensory attribute in fruit (Fawole and Opara, 2013a). Furthermore, Zhang et al. (2009) noted that higher phenolic content observed in dried bitter melon leaves with maturation stages could be attributed to a higher phenolic acid composition at each maturation stage. Besides, the TPC values (101.5, 124.7 and 113.9) at early, commercial and late harvest maturities, respectively, were within the range reported for pomegranate aril (105 and 133 mg GAE/ g) after drying (Thakur et al., 2010) showing optimum retention of TPC after drying. Similarly, as shown in Fig. 3, the TAC of dried arils at commercial maturity was significantly ( $p < 0.05$ ) higher (23.8 mg C3gE/g) than early and late harvest maturities (20.7 and 22.5 mg C3gE/g), respectively. Also, the TAC values in the early, commercial and late harvest maturities are within the range reported for pomegranate aril after drying (20 and 40 mg C3gE/g) (Bchir et al., 2012; Bhat et al., 2014) which shows optimum retention of TAC after drying.

### **3.6. Antioxidant activity**

Radical scavenging assay (RSA) and ferric reducing antioxidant power (FRAP) were used to evaluate the antioxidant capacity of pomegranate dried arils. These assays have been used to assess antioxidant capacity in pomegranate (Fawole and Opara, 2012; Arsende et al., 2014; Mphahlele et al., 2016). In dried arils, significant ( $p < 0.05$ ) highest antioxidant (RSA) values were observed at commercial and late harvest (29.9 and 27.5 mM TE/g), respectively than early harvest maturity (23.4 mM TE/g) (Fig. 4a). However, the highest FRAP activity of dried arils was found in at commercial maturity (3.58 mM TE/g) than early and late harvest (2.41 and 2.84 mM TE/g), respectively, (Fig. 4b). The low antioxidant capacity for dried aril at early harvest could be due to lower phenolic content as the fruit matures. Similar results were reported by Fawole and Opara



(2013b) and Mphahlele et al. (2014) that fruits harvested at whole matured stage resulted in a significantly higher amount of antioxidant capacity than in early harvest.

### 3.7. Principal component analysis and correlation

The results show the average of chemical attributes, phenolic contents, antioxidant activity and colour coordinates of pomegranate dried arils at early, commercial and late harvest maturities. The two principal components (PC1 and PC2) explain 100.0% of the total data variance (Fig. 5). As observed, PC1 explained 92.58% of the total variance while PC2 explained only 7.42% of the total variability which showed that the disparity among dried pomegranate arils as described by the F1 (Fig. 5). The observations (Fig. 5) indicated that dried arils at commercial (H2) and late harvest maturity (H3) could be associated with total soluble solids (TSS), TSS/TA, BrimA, total phenolic content (TPC), total anthocyanin content (TAC), radical scavenging activity (RSA) and ferric reducing antioxidant power (FRAP), chroma ( $C^*$ ) and redness ( $a^*$ ) which had higher positive scores along F1 (Table 4). Also, as observed in Table 5, antioxidant capacity (RSA and FRAP) showed a positive correlation with total phenolic content and total anthocyanin content. This is expected as the increasing consumption of dried pomegranate arils is linked to the high phenolic compounds, which has been reported to highly beneficial to human health (Thakur et al., 2010). Moreover. The higher negative scores (Table 4) along F1 (Fig. 5) correspond to lightness ( $L^*$ ), hue ( $h^\circ$ ), pH and titratable acidity (TA) of the pomegranate dried arils at early harvest (H1). Table 5 showed a significantly ( $p < 0.05$ ) strong relationships were revealed among some of the parameters assessed. For instance, TSS and TA showed a strong negative correlation ( $r = -0.888$ ). This relationship clearly showed that the decrease in fruit titratable acidity might also result in an increase in TSS during fruit development. Another interesting relationship was the strong positive correlation between redness ( $a^*$ ) and total anthocyanin content. This suggests that high total anthocyanin content would contribute to better red colouration of dried arils, which is a desirable attribute in pomegranate marketing. Likewise, high positive scores (Table 4) along F2 is associated with ferric reducing antioxidant power (FRAP) of dried arils at commercial harvest maturity (H2) (Fig. 5). However, lower positive scores along F2 were from dried arils at early harvest ( $L^*$ , hue and pH) and commercial harvest (total phenolic content (TPC) and total anthocyanin content (TAC)). The lower negative scores along F2 (Fig. 5 and Table 4) were at late harvest (H3) (associated with total soluble solids (TSS), BrimA, TSS/TA, titratable acidity (TA), radical scavenging activity (RSA) chroma ( $C^*$ ) and redness ( $a^*$ ). The results demonstrated that PCA



showed that dried products obtained at different harvest times (early, commercial and late) had different quality attributes.

#### **4. Conclusion**

The study has demonstrated that there were significant variations in the harvest maturity stages investigated with regards to the colour, chemical and phytochemical properties of pomegranate dried arils. Based on the retention of TPC, TAC and antioxidant capacity (RSA and FRA), commercial harvest performed better than early and late harvest maturity. The values of titratable acidity, total soluble solids, BrimA and pH observed was optimum in the commercial harvest, which could be used to define flavour of pomegranate dried arils. The evaluated characteristic components reported in this study provides the first scientific evidence that could be used towards quality attributes including physicochemical phytochemical and antioxidant profiling of dried pomegranate arils obtained from different harvest maturity stages in South Africa and could assist in the development of objective indices for processing and quality assessment of dried arils.

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**Table 1**

Quality indices of fresh pomegranate fruit (cv. Wonderful) at different harvest maturities

Attribute	Early (H1)	Commercial (H2)	Late (H3)
Moisture (%)	76.99 ± 0.45 <sup>a</sup>	78.51 ± 1.03 <sup>a</sup>	78.63 ± 0.85 <sup>a</sup>
pH	3.40 ± 0.02 <sup>a</sup>	3.06 ± 0.06 <sup>b</sup>	2.96 ± 0.11 <sup>b</sup>
TA (% citric acid)	1.62 ± 0.01 <sup>a</sup>	1.42 ± 0.03 <sup>b</sup>	1.37 ± 0.01 <sup>c</sup>
TSS (°Brix)	13.2 ± 0.18 <sup>c</sup>	15.8 ± 0.15 <sup>b</sup>	16.2 ± 0.11 <sup>a</sup>
TSS/TA	8.11 ± 0.11 <sup>c</sup>	11.2 ± 0.19 <sup>b</sup>	11.9 ± 0.12 <sup>a</sup>
Weight of fruit (g)	523.6 ± 10.2 <sup>a</sup>	554.2 ± 10.5 <sup>a</sup>	548.6 ± 11.6 <sup>a</sup>
Weight of aril (g)	0.32 ± 0.01 <sup>a</sup>	0.34 ± 0.01 <sup>a</sup>	0.33 ± 0.01 <sup>a</sup>
Aril lightness (L*)	27.6 ± 0.27 <sup>a</sup>	25.4 ± 0.85 <sup>b</sup>	25.2 ± 0.63 <sup>b</sup>
Aril redness (a*)	14.9 ± 0.64 <sup>b</sup>	18.1 ± 0.39 <sup>a</sup>	17.3 ± 0.74 <sup>a</sup>
Aril chroma (C*)	17.8 ± 0.07 <sup>c</sup>	20.7 ± 0.18 <sup>a</sup>	19.6 ± 0.22 <sup>b</sup>
Aril hue (h°)	33.4 ± 0.87 <sup>a</sup>	28.7 ± 0.73 <sup>b</sup>	28.5 ± 0.61 <sup>b</sup>

Titrateable acidity (TA), Total soluble solids (TSS). Data presented as means ± SE in each column followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.

**Table 2**

Chemical properties of dried pomegranate arils obtained from fruit harvested at different maturities

Harvest maturity	TSS (°Brix)	TA (% citric acid)	TSS/TA	BrimA	pH
Early (H1)	14.8±0.28 <sup>b</sup>	1.77±0.03 <sup>a</sup>	8.35±0.18 <sup>b</sup>	11.2±0.28 <sup>b</sup>	3.65±0.01 <sup>a</sup>
Commercial (H2)	20.3±1.06 <sup>a</sup>	1.31±0.06 <sup>b</sup>	15.5±0.46 <sup>a</sup>	17.7±0.98 <sup>a</sup>	3.37±0.02 <sup>b</sup>
Late (H3)	22.2±0.13 <sup>a</sup>	1.42±0.20 <sup>b</sup>	15.8±1.10 <sup>a</sup>	19.4±0.31 <sup>a</sup>	3.33±0.01 <sup>b</sup>

TSS; total soluble solids, TA; titrateable acidity. Data presented as means ± SE in each row followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.

**Table 3**

Colour attributes of dried pomegranate arils obtained from fruit harvested at different maturities

Harvest maturity	L*	a*	C*	h°
Early (H1)	22.4±0.54 <sup>a</sup>	14.5±0.32 <sup>b</sup>	18.1±0.52 <sup>b</sup>	36.3±1.79 <sup>a</sup>
Commercial (H2)	21.0±0.74 <sup>a</sup>	19.4±0.12 <sup>a</sup>	22.3±0.10 <sup>a</sup>	29.3±1.09 <sup>b</sup>
Late (H3)	20.2±1.48 <sup>a</sup>	19.3±0.12 <sup>a</sup>	21.9±0.26 <sup>a</sup>	28.4±0.66 <sup>b</sup>

L\*; lightness, a\*; redness, C\*; chroma, h°; hue angle. Data presented as means ± SE in each row followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.

**Table 4**

Factor loadings and score for the first two principal (F1–F2) components based on dried pomegranate arils at different harvest maturities.

Loadings	F1	F2
pH	-0.990	0.143
TSS	0.955	-0.297
TA	-0.984	-0.177
TSS/TA	0.996	-0.090
BrimA	0.969	-0.247
TPC	0.938	0.347
TAC	0.947	0.320
RSA	1.000	0.011
FRAP	0.807	0.591
L*	-0.917	0.399
a*	0.999	-0.033
C*	1.000	0.014
h°	-0.988	0.154
Scores		
Early (H1)	-4.901	0.066
Commercial (H2)	2.653	1.169
Late (H3)	2.248	-1.235

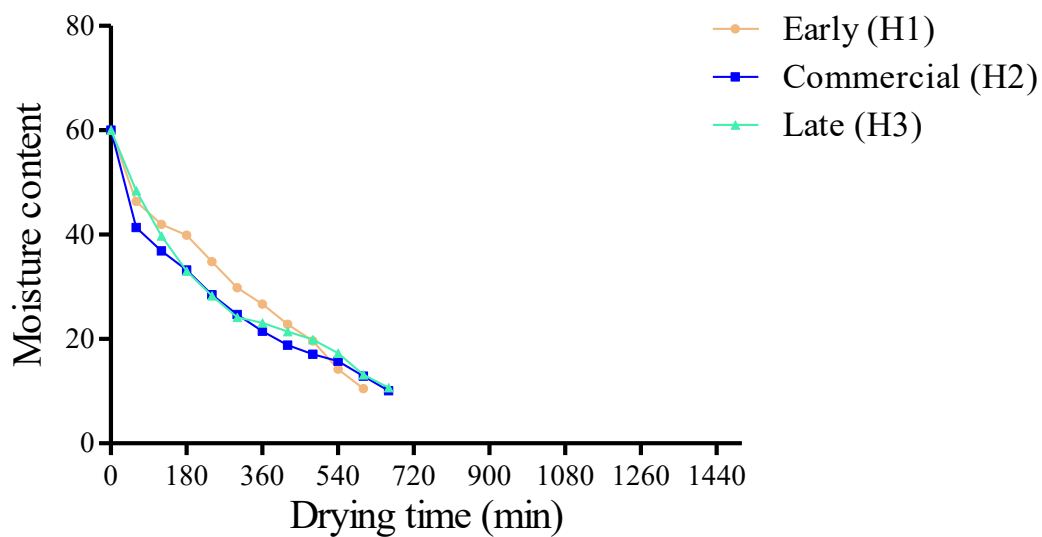
L\*; lightness/darkness, a\*; redness/greenness, C\*; chroma, h°; hue angle, RSA, radical scavenging activity; FRAP, ferric reducing antioxidant power, TPC, total phenolic content; TAC, total anthocyanin content; TSS, total soluble solids; TA, titratable acidity.

**Table 5**

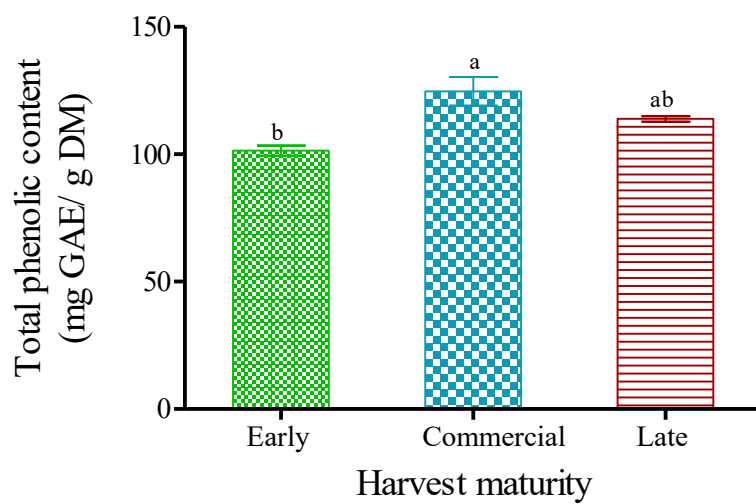
Pearson's correlation coefficients (r) among the investigated parameters of different harvest maturity stages.

Variables	pH	TSS	TA	TSS/TA	TPC	TAC	RSA	FRAP	L*	a*	C*	h°
pH	<b>1</b>											
TSS	-0.988	<b>1</b>										
TA	0.949	-0.888	<b>1</b>									
TSS/TA	<b>-0.999</b>	0.978	-0.964	<b>1</b>								
TPC	-0.878	0.792	-0.984	0.903	<b>1</b>							
TAC	-0.892	0.810	-0.989	0.915	<b>1.000</b>	<b>1</b>						
RSA	-0.988	0.952	-0.986	0.995	0.941	0.951	<b>1</b>					
FRAP	-0.714	0.595	-0.898	0.750	0.962	0.953	0.813	<b>1</b>				
L*	0.965	-0.994	0.832	-0.949	-0.721	-0.741	-0.913	-0.504	<b>1</b>			
a*	-0.994	0.964	-0.978	<b>0.998</b>	0.926	0.936	<b>0.999</b>	0.787	-0.930	<b>1</b>		
C*	-0.988	0.951	-0.987	0.995	0.943	0.952	<b>1.000</b>	0.815	-0.911	<b>0.999</b>	<b>1</b>	
h°	<b>1.000</b>	-0.989	0.945	<b>-0.998</b>	-0.873	-0.887	-0.986	-0.706	0.967	-0.993	-0.986	<b>1</b>

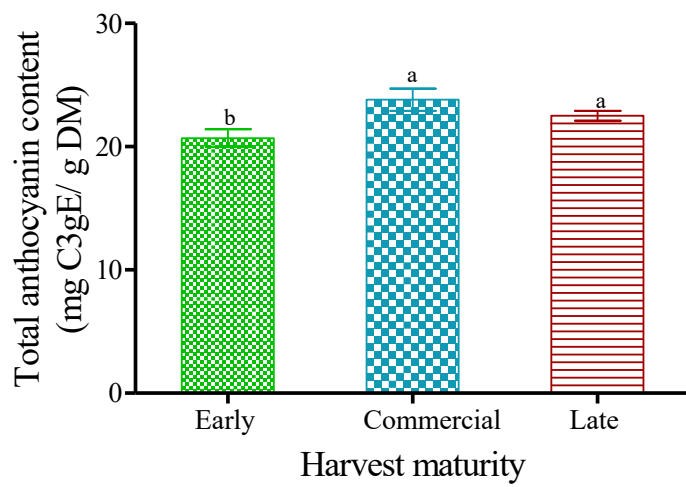
Correlation values in **bold** are significant at  $p < 0.05$ . TPC; total phenolic content, TAC; total anthocyanin content, RSA; radical scavenging activity, FRAP; ferric reducing antioxidant power, L\*; lightness, a\*; redness, C\*; chroma, h°; hue, TSS; total soluble solids, TA; titratable acidity.



**Fig. 1.** Moisture content vs drying time (min) at 60°C, 19.6% relative humidity (RH) of pomegranate dried arils at early (H1), commercial (H2) and late harvest (H3).



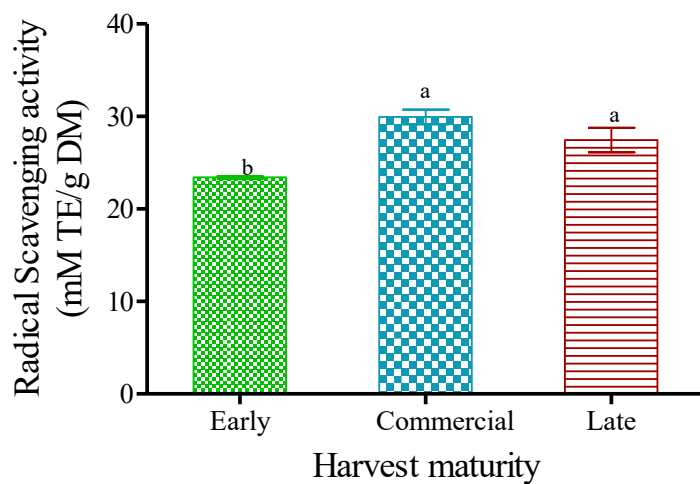
**Fig. 2.** Total phenolic content of dried pomegranate arils obtained at early (H1), commercial (H2) and late harvest (H3). GAE, gallic acid equivalent, DM; dry matter (dried pomegranate arils).



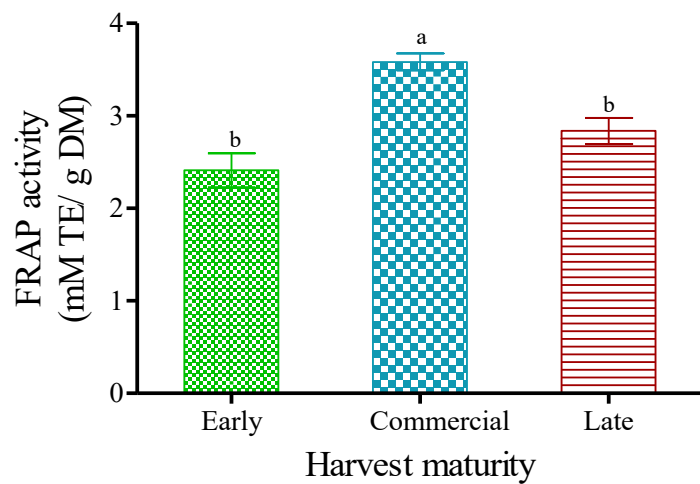
**Fig. 3.** Total anthocyanin content of dried pomegranate arils obtained at early (H1), commercial (H2) and late harvest (H3). C3gE; cyanidin-3glucoside equivalent, DM; dry matter (dried pomegranate arils).



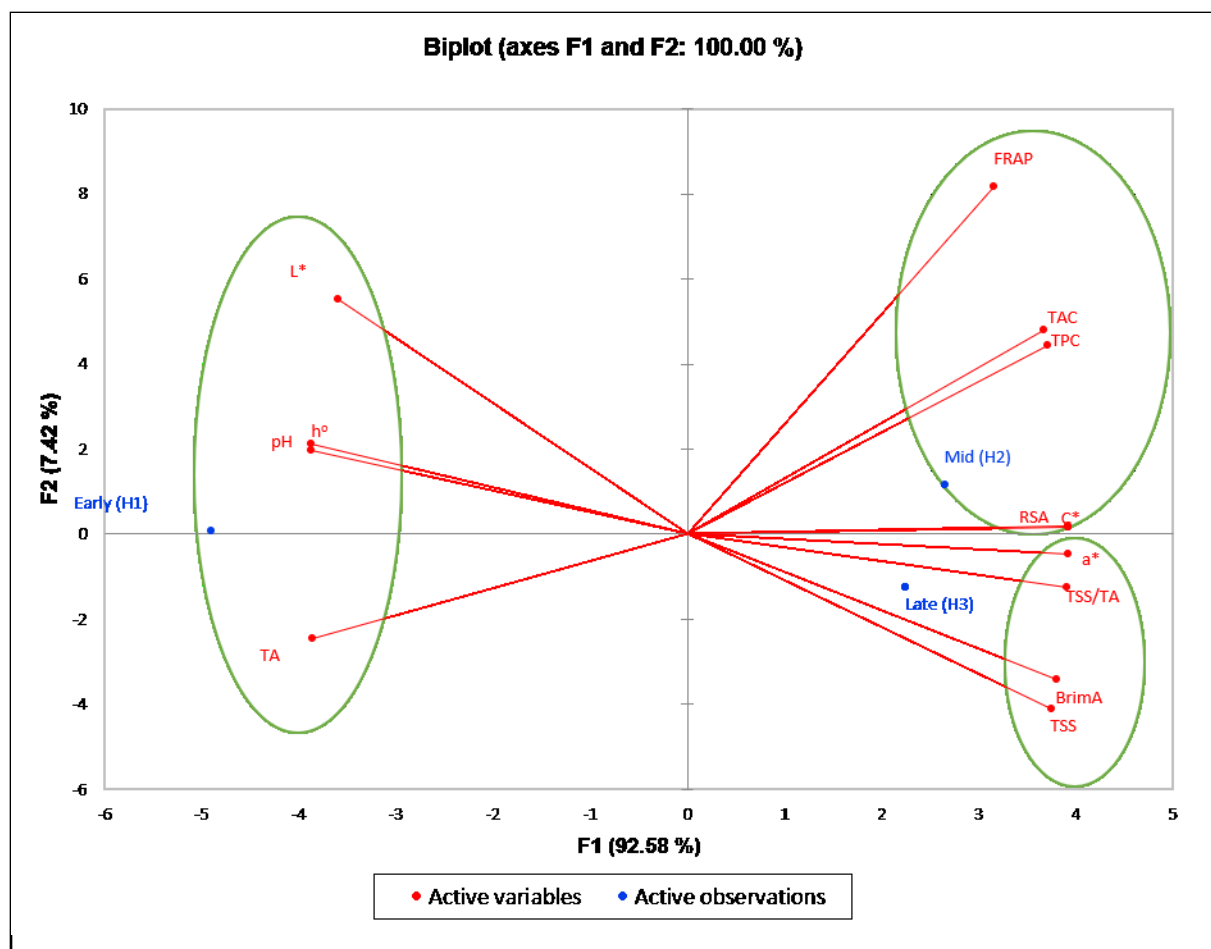
(a)



(b)



**Fig. 4.** Antioxidant capacity (a) RSA and (b) FRAP activity of dried pomegranate arils obtained from fruit harvested at early (H1), commercial (H2) and late harvest (H3). RSA; radical scavenging activity, FRAP; ferric reducing antioxidant power, TE; Trolox equivalent, DM; dry matter (dried pomegranate arils).



**Fig. 5.** Principal component analysis of the first two factors (F1 and F2) based on physicochemical and phytochemical properties as well as antioxidant capacity of dried pomegranate arils cv. Wonderful obtained at different harvest maturity. TSS, total soluble solids; TA, total titratable acidity; TAC, total anthocyanin content, TPC, total phenolic content; RSA, radical scavenging activity; FRAP, ferric reducing antioxidant power.  $L^*$ , lightness;  $C^*$ , chroma;  $h^\circ$ , hue angle;  $a^*$ , redness.

## PAPER 3

### Effect of long-term cold storage of whole fruit on quality attributes of hot-air and freeze-dried pomegranate (*Punica granatum*) arils

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#### ABSTRACT

The effect of cold storage ( $7 \pm 0.3^{\circ}\text{C}$ , with  $92 \pm 3$  % relative humidity) of whole pomegranate fruit on the physicochemical, phytochemical and antioxidant capacity of dried arils was investigated. Extracted arils were dried using hot-air and freeze-drying methods at monthly intervals during 12 weeks of cold storage of whole fruit. Hot-air drying had the least total colour difference (TCD; 3.02) at the end of 12 weeks storage period, while the highest TCD was observed in freeze-dried arils (23.6) at the end of storage. Hot-air dried aril was 46 % higher in total soluble solids (TSS) compared to freeze-dried arils. During the storage of pomegranate fruit, a steady increase in total phenolic content (TPC) from 275.8 to 297.7 mg GAE/100 mL, while total anthocyanin content (TAC) increased from 69.1 to 87.7 mg C3gE /100 mL. Similarly, 9.3 and 5.0 % increase in TPC were observed for both hot-air and freeze-dried arils, respectively, while TAC increased by approximately 13.0 and 5.0 % in hot-air and freeze-dried pomegranate arils, respectively. However, a reduction in the antioxidant capacity in both fresh and dried arils with prolonged storage of whole pomegranate fruit was observed. For instance, the radical scavenging activity (RSA) reduced by 8.5 and 17.4 %, respectively, at the end of the 12 weeks cold storage.

Keywords: Total colour difference, total soluble solids, total phenolic content, antioxidants, storage stability

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#### 1. Introduction

Pomegranate fruit is renowned as a rich source of bioactive phenolic compounds, including flavonoids, phenolic acids, tannins, ellagitannins, catechin, rutin and epicatechin (Fawole et al., 2012; Mphahlele et al., 2016). These antioxidants have been implicated in the protection against diseases related to heart, cancer, immune system and other chronic diseases (Opara et al., 2009; Fawole and Opara, 2013). Recently, the production of pomegranate in South Africa has been estimated to be 540,000 tonnes/1,200,000 cartons, which accounted for the highest portion of the production and export of pomegranate in the Southern hemisphere (Pomegranate Association of South Africa; POMASA, 2018). However, 11 % of the total production is processed locally and 9

% are considered as wastes due to disorders such as cracks, sunburn, scalds and bruises, leading to changes in the internal quality of the fruit (Arsende et al., 2014a; Pomegranate Association of South Africa; POMASA, 2018). In addition, the practicality of storing rawmaterials before processing as applicable to pomegranate, for instance, fruit are harvested within a short window period while processing is carried out over a long period, a situation that requires the storage of rawmaterials mainly for niche product production. Also, 50 % of the fruit consists of the edible part (arils), which are converted into jellies and juices and does not support prolong storage (Mphahlele et al., 2014). Caleb et al. (2013) reported a maximum of 7 days flavour-life of pomegranate arils. However, drying is an option used to preserve food materials by reducing its moisture content and extend the shelf-life of the product (Kingsley and Singh, 2007).

Dried pomegranate arils are often referred to as anardana and are used in many traditional medicinal formulations to cure neurological disorders, stomach and cardiac infections as well as kidney disorders (Jalikop et al., 2002). Dried arils help to improve digestion and mouth-feel due to its high acid content (Singh and Kingsley, 2008), and are used as a condiment especially in Indian and Pakistani cuisines, culinary preparations such as topping for fruit salads, yoghurt and ice cream flavours and as a substitute for tamarind and mango powder (Sharma and Thakur, 2016). However, methods of drying, packaging materials and storage condition are major factors affecting the quality of the final product (Wu et al., 2010).

In addition to the decline in quality of pomegranate fruit during storage, processing also has a negative impact on the fruit quality depending on the techniques used. Based on previous studies, freeze-drying method demonstrated high retention of bioactive compounds during the processing of fruit compared to other drying methods. For instance, Asami et al. (2003) reported higher retention of phenolic concentration in 'Marion' blackberries during freeze-drying than hot-air drying. Shofian et al. (2011) reported that due to the minimal heat treatment applied to remove water from fruit tissue, freeze-drying helped to preserve the antioxidant capacity of tropical fruits. Furthermore, Torres et al. (2010) noted a significant increase in the amount of volatile compounds of grape skin with the freeze-drying compared to the oven-drying method.

In comparison to air drying, freeze-drying improved the retention of bioactive compounds in organic berries during processing and in many cases, increased the concentration of the phytochemicals (Sablani et al., 2011). However, the freeze-drying process could be expensive and

energy-consuming (Ratti, 2001). Among several drying methods available, the hot-air drying method is cost and energy-efficient, making it one of the most frequently used methods for food dehydration (Vega-Galvez et al., 2009). However, it has a greater effect on the deformation of final products which is often characterised by dislocation of volatile substances and changes in physical properties (Lewicki and Jakubczyk, 2004).

‘Wonderful’ pomegranate is the leading pomegranate cultivar grown and consumed globally (Holland et al., 2009; Fawole et al., 2020). In the past years, South Africa has observed a considerable increase in the export production, estimated at approximately 70% of total production (Pomegranate Association of South Africa (POMASA), 2019) unlike 56% in 2013 (Hortgro, 2014). Its bioactive compounds are better maintained with storage temperature and duration compared to other cultivars and thus preferred to other cultivars. For example, longer storage potential up to 5 months was reported while investigating the effect of storability on phytochemical and antioxidant properties of pomegranate fruit (Arendse et al., 2014b). ‘Wonderful’ pomegranate with greater agronomic potential, exhibited the highest scavenging capacity among eight pomegranate cultivars investigated (Fawole and Opara, 2014).

The concentration of bioactive compounds in dried fruit products is dependent on many factors including cultivar, harvest maturity, processing method and storage conditions (Rickman et al., 2007). While there are several studies on the effect of cultivar (Wojdylo et al., 2016; Zhang et al., 2009), information on the impact of harvest maturity and processing method (Beaudry et al., 2004; Sablani et al., 2011) on the bioactive compounds of dried fruit is rare. There is limited information on the preservation of the biochemical components in dried pomegranate aril, particularly considering the phenomena attributed to prolong storage of raw material and processing. Therefore, this study aimed to investigate the effects of prolonged cold storage of whole fruit (raw material) on the quality attributes of dried pomegranate arils. In this present study, the specific processing methods investigated were hot-air and freeze-drying.

## **2. Materials and methods**

### **2.1. Fruit supply and storage condition**

Pomegranate fruit (cv. Wonderful) was handpicked at commercial harvest period from Blydeverwacht orchard in Wellington, (latitude 33°01’00” S, longitude 18°58’59” E) Western

Cape Province, South Africa) during the 2018/2019 growing season. Fruit were transported in an air-conditioned vehicle to the Postharvest Technology Research Laboratory at Stellenbosch University. Fruit free from blemishes and visible external damage were sorted for uniformity of colour and size. After sorting, fresh fruit were packed inside standard open top cartons with the following dimensions: width 0.3 m, length 0.4 m, height 0.133 m and a total of 22 perforations and stored at  $7 \pm 0.3$  °C, with  $92 \pm 3\%$  relative humidity (RH). Fruit were sampled at 0, 4, 8 and 12 weeks as described in the experimental flow chart (Fig. 1). Temperature (°C) and relative humidity (% RH) within the cold rooms were recorded every hour throughout the storage period using Tiny Tag TV-4500 data loggers (Gemini Data Logger, Sussex, UK) with a functional range of  $-40$  °C to  $+85$  °C and 0% to 100% RH.

## **2.2. Characterisation of fresh arils**

Fresh pomegranate arils were periodically evaluated before processing for total soluble solids (TSS) (by refractometric method) and titratable acidity (TA) (by titrating to pH 8.1 with 0.1 N NaOH). Also, moisture content was measured by digital moisture analyser. Total phenolic content (TPC) was determined by the Folin–Ciocalteu method and expressed as mean  $\pm$  SE (milligrams) gallic acid equivalent (GAE) per 100 mL of crude juice while total anthocyanin content (TAC) was quantified using the pH differential method (Wrolstad, 1993) and expressed as mean  $\pm$  SE (milligrams) cyanidin-3-glucoside per 100 mL of crude juice. The antioxidant capacity (radical scavenging activity, RSA; ferric ion reducing antioxidant power, FRAP) was also measured in triplicate, according to Fawole and Opara (2012) and expressed as Trolox equivalent (mM) per 100 mL of crude juice.

## **2.3. Drying procedure**

### **2.3.1. Freeze-drying**

Arils were placed in a freeze-drying paper bag and frozen in a static air freezer at  $-80$  °C. Freeze-drying of frozen samples was carried out in triplicates. Specimen jar containing samples were carefully taken to a laboratory-scale freeze-dryer (VirTis Co., Gardiner, NY, USA) operating at condenser temperature  $-85$ °C and pressure 6 Pa. Weight loss was recorded at 3 h interval and the drying time to reach equilibrium weight was 96 h.

### 2.3.2. Hot-air drying

Arils were dried at 60 °C in a hot-air oven for 11 h to achieve a 10-12 % moisture content. Constant air velocity was maintained at 1.0 m s<sup>-1</sup> for each treatment. The hot-air dryer was kept functional for an hour to equilibrate the inner temperature before drying. The accuracy of the inner temperature was monitored using a thermometer before aril was spread in glassy Petri dishes and placed in the drying chamber. Dried arils were packed and sealed in food-grade moisture-resistant plastic bags and stored in glass desiccators containing calcium sulfate (Sigma-Aldrich Pty. Johannesburg, South Africa).

### 2.4. Colour measurement

Dried aril colour was determined by direct reading using a chromo-meter (Minolta model CR-200, Osaka, Japan) to obtain the colour values:  $L^*$  (brightness/darkness),  $a^*$  (redness/greenness),  $C^*$  (colour intensity) and  $h^\circ$  (colour purity). The measurements were taken at three different times from a transparent petri dish and averaged. The maximum for  $L^*$  value is 100 (white), and the minimum is zero (black). Positive  $a^*$  value is red, negative  $a^*$  is green, while positive  $b^*$  value is yellow and negative  $b^*$  is blue. The total colour difference (TCD) were calculated (Fawole et al., 2012).

$$\text{TCD} = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (1)$$

Where  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$  represents the value before and after drying at each treatment levels and results were expressed as means  $\pm$  S.E.

### 2.5. Characterisation of dried arils

Dried pomegranate arils were ground into powder using liquid nitrogen followed by extraction of 5 g sample in 50 mL distilled water. The mixture was vortexed for 5 min and sonicated for 15 min in an ultrasonic bath. This was followed by centrifugation at 10 000 rpm for 25 min and recovery of the supernatant for TSS, TA and pH measurements. For phytochemical properties and antioxidant capacity, the same extraction procedure was followed using 50 % methanol.

## 2.6. Chemical properties

### 2.6.1. Total soluble solids and titratable acidity determination

TSS was measured using a digital hand refractometer (model PT-32; ATAGO, Tokyo, Japan) with the range of 0–32 °Brix which was blanked with distilled water. For TA, two millilitres of supernatant was diluted in 70 mL of distilled water and titrated against 0.2 N of sodium hydroxide (NaOH) to a pH of 8.2 using a Metrohm 862 Compact titrosampler (Herisau, Switzerland).

## 2.7. Determination of phytochemical properties

### 2.7.1. Total phenolic content (TPC)

TPC of dried arils was determined by the Folin–Ciocalteu method using a methanolic extract of dried arils (Fawole et al., 2012). The supernatant (0.05 mL) was mixed with 0.45 mL of 50 % methanol in a test tube followed by the addition of 0.5 mL Folin–Ciocalteu after 2 min. The mixture was then vortexed and kept in the dark for 10 min before adding 2 % Na<sub>2</sub>CO<sub>3</sub> and further incubated for 40 min in the dark. The absorbance of each sample was read at 520 nm in a UV-visible spectrophotometer (Thermo Scientific technologies, Madison, USA) against a blank containing 50 % methanol. Absorbance was compared with a standard curve (Gallic acid, 0 – 10 mg), and results were expressed as mg gallic acid equivalent per gram pomegranate dry matter (mg GAE/g DM).

### 2.7.2. Total anthocyanin content

Total anthocyanin content (TAC) was quantified using the pH differential method (Wrolstad, 1993). In triplicates, extract (1 mL) was mixed with 9 mL of pH 1.0 and pH 4.5 buffers separately. Absorbance was measured at 520 and 700 nm in pH 1.0 and 4.5 buffers, and the result was expressed as cyanidin 3-glucoside using equation 2.

$$A = (A_{510} - A_{700})_{pH\ 1.0} - (A_{510} - A_{700})_{pH\ 4} \quad (2)$$

$$\text{Total monomeric anthocyanin (mg/mL)} = \frac{A \times MW \times DF}{\epsilon \times L}$$

where A = Absorbance,  $\epsilon$  = Cyd-3-glucoside molar absorbance (26,900), MW = anthocyanin molecular weight (449.2), DF = dilution factor, and L = cell path length (1 cm). Results are expressed as equivalent per gram dry matter (mg C3gE/g DM).



## 2.8. Antioxidant capacity

### 2.8.1. Radical-scavenging activity (RSA)

The RSA was carried out in triplicate, according to Fawole et al. (2012). Briefly, under dim light, aqueous methanolic extract of dried aril (0.015 mL) was diluted with methanol (0.735 mL) in test tubes followed by the addition of methanolic DPPH solution (0.75 mL, 0.1 mM). The mixtures were incubated at room temperature for 30 min in the dark, and the absorbance was measured at 517 nm using a UV-vis spectrophotometer. Absorbance was compared with the standard curve (Trolox equivalent, 0 – 2.0 mM). The free-radical activity of dried aril was expressed as Trolox equivalent (mM) equivalent per gram dry matter (mM TE/g DM).

### 2.8.2. Ferric ion reducing antioxidant power (FRAP)

The antioxidant power of dried aril was measured using the colourimetric method according to Benzie and Strain (1996) and Fawole et al. (2013). The FRAP working solution was freshly prepared in mixtures of 300 mM acetate buffer (50 mL), 10 mM 2,4,6-tripyridyl-s-triazine (TPTZ) (5 mL) and 20 mM ferric chloride (5 mL) at 37 °C. In triplicates, diluted aqueous methanolic dried aril extracts (0.15 mL) were added to 2.85 mL of the FRAP working solution before incubation in the dark for 30 min. The reduction of the  $\text{Fe}^{3+}$ -TPTZ complex to a coloured  $\text{Fe}^{2+}$ -TPTZ complex at low pH by dried aril extracts was monitored by measuring the absorbance at 593 nm. Trolox (0 - 10 mM) was used for the calibration curve, and the results were expressed as Trolox (mM) equivalents per gram dry matter (mM TE/g DM).

### 2.8.3. Stability of RSA and FRAP

Several studies have reported the use of simple first-order reaction kinetics to describe storage and thermal degradation of bioactive compounds from various sources. Li et al. (2012) and Moldovan et al. (2016) described the degradation kinetics as in equation 3:

$$\ln[\text{RSA}] = \ln[\text{RSA}_0] - kt \quad (3)$$

where: RSA = antioxidant capacity, mM TE/g dried aril at time  $t$ ;  $\text{RSA}_0$  = initial RSA, mM TE/g;  $k$  = reaction rate constant,  $\text{weeks}^{-1}$ ;  $t$  = reaction time, weeks. The half-life of antioxidant capacity from the investigated extracts during storage can be calculated using equation (4):

$$t_{1/2} = -\ln 0.5/k \quad (4)$$

where:  $t_{1/2}$  = half-life (weeks);  $k$  = reaction rate constant ( $\text{weeks}^{-1}$ ).

## 2.9. Statistical analysis

The measurement made from chemical properties, colour and phytochemical properties were subjected to statistical evaluation. Data were processed with STATISTICA (Statistica 13.0, StatSoft Inc., Tulsa, OK, USA) and presented as means  $\pm$  standard error. All analysis was done in triplicates. Data was subjected to two-way analysis of variance (ANOVA), and means were separated according to Fisher's LSD test at a level of significance of 95 %. GraphPad Prism software 4.03 (GraphPad Software, Inc., San Diego, USA) was used for graphical presentations. Pearson's correlation was carried out using the XLSTAT software version 2012.04.1 (Addinsoft, France).

## 3. Results and discussion

### 3.1. Effect of cold storage on moisture content of pomegranate aril

From Table 1, a gradual reduction with time was observed in the moisture content of pomegranate aril at 12 weeks storage period. At 8 weeks of storage, visual browning of 5 % was observed in the arils immediately after peeling the fruit, with a gradual increase to 15 % at the end of storage. However, in the pictorial representation, there were no noticeable differences in the arils with storage time, as shown in Fig. 2. These results are similar to the study by Konopacka and Plocharski (2001) who reported increasing tissue browning in apple subjected to long term storage. However, a non-browning condition in aril tissue was reported in the pre-storage chemical dipping of 'Taify' pomegranate fruit (Awad et al., 2013). This could be due to treatment application on the fruit before cold storage. Further, the moisture content of fresh pomegranate arils decreased with storage time from 74.7 to 57.4 % (Table 1), which affects the weight of the fruit. The reduced weight observed during storage could be attributed to the transpiration that occurred due to large pore spaces in the fruit peel, which enhances the permeability of moisture (Fawole and Opara, 2013; Arendse et al., 2015). Moreover, pomegranate fruit has been reported to be highly susceptible to weight loss (Artes et al., 1996) which could lead to shriveling observed in Fig. 2. Additionally, the reduction in the weight of the whole fruit consequently resulted in a weight reduction of the arils. These findings were corroborated in the study by Fawole and Opara (2013), who reported a significant reduction in weight of pomegranate fruit during cold storage.

### 3.2. TCD of fresh and dried pomegranate arils

Storage of pomegranate fruit contributed to the changes in the TCD of fresh arils, and also had a significant effect on the TCD of dried arils. A notable variation was observed in the TCD

with increased storage period, with the highest TCD being 11.2 at the end of storage (Fig. 3). For dried arils processed with hot-air and freeze-dryers, there was a significant ( $p < 0.0001$ ) interaction in TCD (Fig. 4). Hot-air drying had the least TCD (3.02) at the end of 12 weeks storage period, while the highest was observed in freeze-dried arils at the end of storage (23.6) (Fig. 4). The change in TCD is an important part of a dried product, which express human eye capacity to visualise various colours attributed to different products (Wojdylo et al., 2016). Similar to the findings in this study, Coklar et al. (2018) reported a better colour appearance of freeze-dried hawthorn fruit than oven and microwave dryers. Ali et al. (2016) reported that freeze-dried guava fruit had the best colour preservation compared to sunlight and convective oven dryer. Further, the colour change described for dried arils could be influenced partly, by drying methods and also the changes observed in the naturally occurring biochemicals during storage of pomegranate fruit.

### **3.3.Total soluble solids (TSS) and titratable acidity (TA) of fresh and dried arils**

The investigated chemical attributes (TSS and TA) in the fresh aril of pomegranate were significantly ( $p < 0.05$ ) different from the storage period. For instance, The TSS of fresh aril increased from 13.7 to 15.1 °Brix at the end of storage (Table 1), while the TA decreased from 0.38 to 0.24 at 12 weeks storage. In agreement with our study, Arendse et al. (2014) reported that pomegranate cultivar Wonderful stored at 5 °C showed an increase in TSS as the storage period progressed. A decrease in TA could be attributed to organic acid break down during the storage period since pomegranate is a non-climacteric fruit (Kader et al., 1984). Fawole and Opara (2013) observed a decrease in TA values for two South African grown cultivars Bhagwa and Ruby during storage due to the ongoing metabolism in the fruit during storage.

In dried arils processed with hot-air and freeze-dryers, all chemical attributes showed significant ( $p < 0.0001$ ) interactions with storage period and drying methods (Table 2). TSS values gradually increased with storage period in the hot-air dried arils, while a significant decrease in TSS was observed in freeze-dried arils at the end of storage. Moreover, hot-air dried pomegranate arils showed higher TSS values compared to freeze-dried arils. For instance, at the end of storage, hot-air dried aril was 46 % higher in TSS than in freeze-dried arils. The high TSS value could be attributed to the method of drying since hot-air arils involved drying under high temperature, which resulted in the caramelisation of the product (Vanhel and Blond, 1999). Furthermore, the increase in the TA values was observed in arils processed with hot-air compared to freeze-dried arils (Table

2). TA values in hot-air dried arils increased at 4 weeks of storage, and gradually decreased with prolonged storage, while in freeze-dried arils, a slight increase in TA up to 8 weeks was observed followed by a decline at 12 weeks of storage. TA values ranged between 3.10 - 3.15 in hot-air dried arils and 1.14 - 1.24 in freeze-dried arils. The changes observed in the values of TA of dried arils could be attributed to the drying method since there are differences in the drying temperatures for hot-air and freeze-dryers. Ashebir et al. (2009) noted a significant change in the TSS and TA concentrations of dried tomatoes due to variations in the level of drying temperatures.

TSS/TA ratio is one of the quality indexes of pomegranate fruit (Al-Said et al., 2009). An increase in TSS/TA value was observed in the hot-air dried arils at the end of storage, while in freeze-dried arils, a decrease in TSS/TA value was observed at the end of storage (Table 2). This implies that during storage, dried arils with hot-air dryer synthesise more sugar. TSS/TA values ranged between (7.0 and 7.58) in hot-air dried arils and (10.2 – 15.4) in freeze-dried arils. Higher TSS/TA values observed in freeze-dried arils compared to hot-air dried arils could be appropriated to a higher percentage of sugar to acid ratio in dried aril.

### **3.4.Total phenolic content (TPC) and total anthocyanin content (TAC) of fresh and dried arils**

As shown in Table 3, Fig. 3 and 4, long-term storage contributed to the increase in the TPC and TAC, respectively, for fresh and dried pomegranate arils. For instance, during the storage of pomegranate fruit, a steady increase in TPC from 275.8 to 297.7 mg GAE/100 mL, while TAC increased from 69.1 to 87.7 mg C3gE /100 mL (Table 3). This is an indication of the occurrence of the biochemical reaction during storage. Similar to our findings, Arendse et al. (2014) reported an increase in TPC of pomegranate arils cv. Wonderful stored at 5 °C, 7.5 °C, and 10°C for 5 months. The authors reported that the increase in TPC could be related to the continued accumulation of anthocyanins at lower temperatures occurring during storage. In addition, Labbe et al. (Labbe et al., 2010) reported an increase in the total phenolic content of ‘Chilean Chaca’ pomegranate cultivar at 5 °C for 12 weeks.

Moreover, anthocyanin compounds exhibit the main characteristic red colour in pomegranate fruit (Artés et al., 1996). Increase in anthocyanin concentration during storage could be related to the increase in biosynthesis and accumulation of anthocyanin, which is usually induced at lower temperatures in pomegranate fruit (Miguel et al., 2008). Results from this study

agree with those reported by Arendse et al. (2014), who reported an increase in total anthocyanin concentration in pomegranate ‘Wonderful’. In dried pomegranate arils, drying methods contributed to the retention of TPC ( $p < 0.0001$ ) as shown in Fig. 5, while a combined effect of drying method and storage period influenced the TAC ( $p < 0.0001$ ; 0.003, respectively) (Fig. 6). Although there was an increase that occurred in the 12 weeks storage period, there was no difference statistically. At the end of 12 weeks cold storage, TPC increased from 105.9 to 116.7 mg GAE/g DM in hot-air dried pomegranate arils, while in freeze-dried arils (135.6 to 142.7 mg GAE/g DM). The increase of TPC in both hot-air and freeze-dried arils was 9.3 and 5.0 %, respectively, at the end of storage. This is an indication that the influence of cold storage of pomegranate fruit contributed to the retention of TPC of dried arils. Furthermore, the freeze-drying method increased TPC of the dried arils by approximately 18.2 % higher than the TPC obtained from hot-air dried arils. This confirms the study by Shishegharha et al. (2002) who reported that the freeze-drying method is a precision technology utilised to produce high-quality dried products. Additionally, the increased TPC in freeze-dried pomegranate arils could be attributed to mild fruit cell destruction during freezing and ice sublimation which consequently result in increased extraction of biochemical components (Asami et al., 2003).

From Fig. 6, the trend showed an increased TAC of hot-air and freeze-dried pomegranate arils throughout the 12 weeks storage period. This could be as a result of the increasing trend observed in fresh arils at cold storage. At the end of the storage period, TAC increased by approximately 13 % in hot-air dried pomegranate arils, while in freeze-dried arils, an increase of 5 % was observed. However, an increase in the TAC of freeze-dried arils was observed compared to hot-air dried arils. The study by Wu et al. (2010) reported that the increase in the anthocyanin content of freeze-dried blueberry than hot-air dried fruit, and this was reported to be due to the low temperature and vacuum in freeze-drying that preserved the bioactive compounds from oxidation. Freeze-dried blueberries had higher retention of total anthocyanins compared to hot-air dried blueberry because freeze-drying method operates on vacuum pressure and minimal temperature (Mejia-Meza et al., 2008).

### **3.5. Antioxidant capacity of fresh and dried arils**

The antioxidant capacity (RSA and FRAP) of fresh pomegranate arils decreased significantly with storage time. RSA showed a decrease with prolonging storage time from 124.1

to 49.2 mM TE/100 mL (Table 3). Similarly, FRAP decreased from 23.6 to 20.7 mM TE/100 mL (Table 3). In relation to their nutritional benefits, the more the phenolic compounds in the fruit, the more the total antioxidant capacity and its relative human health benefits (Tzulker et al., 2007). The observed increase in both TPC and TAC was the inverse of the trends observed in the antioxidant capacity (RSA and FRAP) exhibited by pomegranate fruit during storage at  $7 \pm 0.3^{\circ}\text{C}$ , with  $92 \pm 3\%$  RH. This suggested that antioxidants often react differently depending on the type of antioxidant assays (Cam et al., 2009). Siddhuraju et al. (2002) reported that the decrease in the reducing power could be attributed to the bioactive compounds, that is, total phenolics, flavonoids, ascorbic acids and other hydrophilic antioxidants associated with the component of the antioxidants present in the fruit.

For dried pomegranate arils, there were significant interactions on the antioxidant capacity (RSA and FRAP) ( $p < 0.023$ ; 0.0001), respectively, (Fig. 7 a and b). The trend showed a general decrease in antioxidants RSA and FRAP for both hot-air and freeze-dried arils at the end of storage. During the storage period, RSA values for hot-air dried arils were within the range of 26.9 and 29.5 mM TE/g DM which was close to the values reported for hot-air dried pomegranate (22.7 to 30.6 mM TE/g) (Golukcu, 2014) and higher than papaya (9.72 mM TE/g) (Chong et al., 2013). On the other hand, FRAP value was maintained at 4 weeks for freeze-dried arils followed by a decline with the storage period. FRAP values in freeze-dried arils were between 2.49 and 3.27 mM TE/g DM, also close to the value reported for freeze-dried pomegranate arils cv. Mollar de Elche (3.4 mM TE/g) (Cano-Lamadrid et al., 2017).

Further, at the end of the storage period, freeze-drying resulted in approximately 12.1 and 22.9 % reduction in antioxidant capacity (RSA and FRAP), respectively, in comparison to hot-air dried arils. The decrease in the antioxidant capacity in dried arils could be attributed to the observed decrease in the antioxidant capacity in fresh arils. This could be partly, due to the decrease observed in the RSA and FRAP of fresh arils during the storage period and rather the thermal degradation of heat-sensitive phenolics since TPC is reported to be the major contributors to antioxidant capacity. Moser et al. (2017) also reported up to 25 % reduction in antioxidant capacity in grape powder after 45 days of storage due to the formation of antioxidant polymers, such as low molecular weight procyanidins. Mrad et al. (2012) noted that ‘decrease in the content of phytochemical compounds during drying could also be attributed to the alterations in the chemical structure of polyphenols.

Similarly, Fracassetti et al. (2013), studying the storage of freeze-dried wild blueberry powder, observed a decline in antioxidant activity. However, Michalczyk et al. (2009) reported that the antioxidant capacity of dried berries was retained with prolonging storage to a very high degree which is in contrast with the results from this present study. As observed in Fig. 7 a and b, hot-air dried arils showed higher antioxidant capacity than freeze-dried arils. Similar to the results from this study, Mphahlele et al. (2016) reported better retention of antioxidants in the oven drying at higher temperatures 60 °C than in freeze-dried pomegranate peel. The authors attributed this to the concentration of compounds contained in the peel, ‘since they act as scavengers of free radicals produced during oxidation reaction’.

### 3.6. Stability of antioxidant capacity (RSA and FRAP) of dried pomegranate arils

Understanding the stability or degradation mechanisms of food products is essential to maximise the nutritional and sensory quality of products (Moser et al., 2017). The stability of antioxidant capacity (RSA and FRAP) of pomegranate arils after hot-air and freeze-drying were evaluated based on changes in their concentrations (Table 4; Fig. 8a, b). Table 4 shows the kinetic parameters (kinetic rate constants and the half-life values) determined for the thermal degradation of the antioxidant capacity. Lower the degradation rate indicates lower kinetic rate constants ( $k$ ) and higher half-life (Moser et al., 2017).

According to Eq. (5), a series of kinetic rate constants ( $k$ ) for both drying methods were obtained by plotting the changes in the antioxidant capacity of pomegranate arils obtained from hot-air and freeze-drying as a function of time (Fig. 8a, b). The RSA activity in freeze-dried arils had lower degradation rate ( $k = 0.146$ ;  $t_{1/2} = 5.844$ ) than hot-air dried arils ( $k = 0.151$ ;  $t_{1/2} = 5.654$ ), however, the FRAP activity in hot-air dried arils had lower degradation rate ( $k = 0.129$ ;  $t_{1/2} = 7.306$ ) than the freeze-dried arils ( $k = 0.143$ ;  $t_{1/2} = 6.597$ ). Considering the calculated degradation  $k$  and  $t_{1/2}$  as an indicator of the amount of antioxidant loss, with a half-life ( $t_{1/2}$ /week), the stability of RSA in the hot-air dried arils was approximately 3.3 % lower than freeze-dried arils. However, the stability in FRAP activity in freeze-dried arils was 9.7 % lower than the hot-air dried arils (Table 4). Several researchers have reported a decrease in the bioactive compounds in fruit after drying (Di Scala and Crapiste, 2008; Vega-Galvez et al., 2009; Devic et al., 2010). Zhou et al. (2016) reported high degradation in antioxidant capacity (DPPH, FRAP and ABTS) of red pepper. Similarly, Garau et al. (2007) also found that the antioxidant capacity in orange peel and pulp both



decreased by air-drying. This is also consistent with the results of this study. More so, the values of coefficients of determination ( $R^2$ ) ranging from 0.85 - 0.99 were obtained for all linear regressions, indicating that the degradation process of these bioactive compounds for both drying hot-air and freeze-drying methods followed first-order reaction kinetics.

### **3.7. Correlations amongst quality attributes for both hot-air and freeze-dried arils at 12 weeks storage period**

Significant relationships that exist among attributes measured for hot-air and freeze-dried arils are presented in Tables 5 a and b. Pearson's correlation tests indicated a strong positive relationship between TPC and TAC ( $r = 0.998$ ) (Table 5a). Also, there were strong negative correlations between TPC and RSA ( $r = -0.894$ ) as well as TPC and FRAP values ( $r = -0.998$ ); TAC and RSA ( $r = -0.910$ ) as well as TAC and FRAP ( $r = -1.000$ ). However, a positive correlation was found between RSA and FRAP ( $r = 0.919$ ) (Table 5a). A similar result was reported by Cano-Lamadrid et al. (2017) between antioxidant ABTS and FRAP in osmotically dehydrated pomegranate arils cv. Mollar de Elche. Strong correlations were found between TSS and phytochemical properties (TPC and TAC), but none of the relationships seems to be applicable in practice. For instance, a strong correlation ( $r = 0.937$ ) was found between TSS and TAC in hot-air drying (Table 5a). In practice, no relevant prediction of dried aril flavour could be made using total anthocyanin content since soluble solids measurement technique applies only to the sweetness ( $^{\circ}\text{Brix}$ ) of aril tissues. However, in hot-air drying, a moderately negative correlation was observed between TSS and TA ( $r = -0.555$ ) (Table 5a). This relationship clearly showed that the increase in total soluble solids of dried arils could also contribute to a decrease in TA. Other relationships found a moderately negative correlation was between TA and TSS/TA (Table 5b).

However, a negative and weak correlation was found between TSS and TA ( $r = -0.304$ ) in freeze-drying (Table 5b). Considering the reported benefits of consuming fruit with high phytochemical properties, it is therefore not surprising that antioxidant capacity (RSA and FRAP) showed a strong positive correlation with TPC. Therefore, the overall quality of dried arils investigated with each of the drying methods clearly showed that only the interactions among the bioactive components seem promising and practicable.



#### 4. Conclusion

Prolong cold storage of raw material considerably affected the total soluble solids and titratable acidity of hot-air and freeze-dried pomegranate arils. Increase in the TSS of fresh arils was noticed while TA decreased with storage period. Freeze-dried aril had a significantly higher total colour difference (TCD) than hot-air dried arils at the end of the storage period. Hot-air dried arils presented the highest TSS and TA compared to freeze-drying at the end of the storage period. A steady increase in the total phenolic content (TPC) and total anthocyanin content (TAC) of both fresh arils and final products was observed at the end of the storage period. Cold storage had negative effects on the antioxidant activity (RSA and FRAP) in both fresh arils and final products. At the end of the storage period, freeze-drying presented higher stability of antioxidant capacity (RSA) than observed in hot-air drying. In contrast, hot-air drying presented higher stability of antioxidant capacity (FRAP) with the highest half lifetime, suggesting that the preservation of antioxidant capacity in dried arils is dependent on the type of assay and choice of drying method. Similarly, due to the reduction in the colour and antioxidant capacity of dried arils with prolonged storage of raw material, fresh pomegranate fruit are suggested for immediate processing after harvest.

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**Table 1**Changes in physicochemical attributes of fresh pomegranate aril during 12 weeks cold storage at  $7 \pm 0.3^\circ\text{C}$ ,  $92 \pm 3\%$  RH (w.b)

Storage period (weeks)	Moisture content (%)	TSS ( $^\circ\text{Brix}$ )	TA (% citric acid)	TSS/TA
0	$74.7 \pm 1.25^a$	$13.7 \pm 0.25^c$	$0.38 \pm 0.03^a$	$36.7 \pm 2.01^c$
4	$71.9 \pm 0.92^a$	$14.4 \pm 0.22^b$	$0.33 \pm 0.01^{ab}$	$44.2 \pm 2.25^c$
8	$67.8 \pm 0.73^b$	$14.8 \pm 0.05^{ab}$	$0.28 \pm 0.01^{bc}$	$53.2 \pm 2.10^b$
12	$57.4 \pm 1.08^c$	$15.1 \pm 0.06^a$	$0.24 \pm 0.01^c$	$62.5 \pm 2.97^a$

TSS, total soluble solids; TA, titratable acidity. Data presented as means  $\pm$  SE in each column followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.

**Table 2**Changes in the chemical properties of dried pomegranate arils during 12 weeks cold storage at  $7 \pm 0.3^\circ\text{C}$ ,  $92 \pm 3\%$  RH (w.b)

Drying method	Storage period (weeks)	TSS ( $^\circ\text{Brix}$ )	TA (% Citric acid)	TSS:TA
Hot-air drying	0	$22.2 \pm 0.67^a$	$3.15 \pm 0.17^b$	$7.03 \pm 0.19^c$
	4	$22.7 \pm 0.73^a$	$3.23 \pm 0.01^a$	$7.00 \pm 0.21^c$
	8	$23.7 \pm 0.44^a$	$3.13 \pm 0.00^{bc}$	$7.55 \pm 0.15^c$
	12	$23.5 \pm 0.58^a$	$3.10 \pm 0.02^c$	$7.58 \pm 0.22^c$
Freeze-drying	0	$17.5 \pm 1.00^b$	$1.14 \pm 0.01^c$	$15.4 \pm 0.86^a$
	4	$15.0 \pm 0.29^c$	$1.20 \pm 0.01^d$	$12.5 \pm 0.36^b$
	8	$14.0 \pm 0.50^{cd}$	$1.24 \pm 0.03^d$	$11.3 \pm 0.62^b$
	12	$12.8 \pm 0.33^d$	$1.14 \pm 0.01^c$	$10.2 \pm 0.36^b$
Drying method (A)		0.0001	0.0001	0.0001
Storage period (B)		0.091	0.0001	0.002
A x B		<b>0.0007</b>	<b>0.006</b>	<b>0.0002</b>

TSS, total soluble solids; TA, titratable acidity. Data presented as means  $\pm$  SE in each row followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.

**Table 3**Changes in the phytochemical properties and antioxidant capacity of fresh pomegranate arils during 12 weeks cold storage at  $7 \pm 0.3^{\circ}\text{C}$ ,  $92 \pm 3\%$  RH (w.b.)

Storage period (weeks)	TPC mg GAE/100 mL	TAC cyaniding-3-glucoside (mg/100 mL)	RSA mM TE/100 mL	FRAP mM TE/100 mL
0	275.8 $\pm$ 5.86 <sup>c</sup>	69.1 $\pm$ 3.11 <sup>c</sup>	124.1 $\pm$ 1.66 <sup>a</sup>	23.6 $\pm$ 0.36 <sup>a</sup>
4	287.3 $\pm$ 0.53 <sup>b</sup>	75.6 $\pm$ 4.88 <sup>bc</sup>	103.6 $\pm$ 1.66 <sup>b</sup>	22.7 $\pm$ 0.05 <sup>a</sup>
8	294.5 $\pm$ 1.96 <sup>ab</sup>	84.4 $\pm$ 1.62 <sup>ab</sup>	83.5 $\pm$ 1.71 <sup>c</sup>	20.9 $\pm$ 0.34 <sup>b</sup>
12	297.7 $\pm$ 2.32 <sup>a</sup>	87.7 $\pm$ 0.37 <sup>a</sup>	49.2 $\pm$ 3.79 <sup>d</sup>	20.7 $\pm$ 0.68 <sup>b</sup>

RSA, radical scavenging activity; FRAP, ferric reducing antioxidant power; TPC, total phenolic content; TAC, total anthocyanin content; w.b. wet basis; Data presented as means  $\pm$  SE in each column followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.



**Table 4**

Effect of drying methods on the kinetic parameters of antioxidants (RSA and FRAP) degradation in dried pomegranate arils

Drying methods	Antioxidant (mM TE/g)	$k \times 10^{-3}/(\text{week}^{-1})$	$t_{1/2}/\text{week}$	$R^2$
Hot-air	RSA	0.151	5.654	0.9949
	FRAP	0.129	7.306	0.9949
Freeze-drying	RSA	0.146	5.844	0.9031
	FRAP	0.143	6.597	0.8582

$k$ , kinetic rate constants;  $t_{1/2}$ ; half-life;  $R^2$ , coefficients of determination; RSA, radical-scavenging activity; FRAP, ferric ion reducing antioxidant power.

**Table 5a**

Pearson's correlation coefficients among the investigated parameters of hot-air dried pomegranate arils for 12 weeks storage period

Variables	TCD	TSS	TA	TSS:TA	TPC	TAC	FRAP	RSA
TCD	1							
TSS	0.052*	1						
TA	0.687**	-0.555*	1					
TSS:TA	-0.237 <sup>ns</sup>	0.941**	-0.804**	1				
TPC	0.067 <sup>ns</sup>	0.946**	-0.649**	0.944**	1			
TAC	0.122 <sup>ns</sup>	0.937**	-0.612**	0.922**	0.998**	1		
FRAP	-0.132 <sup>ns</sup>	-0.944**	0.599*	-0.922**	-0.998**	-1.000**	1	
RSA	0.430 <sup>ns</sup>	-0.905**	0.244 <sup>ns</sup>	-0.749**	-0.894**	-0.910**	0.919**	1

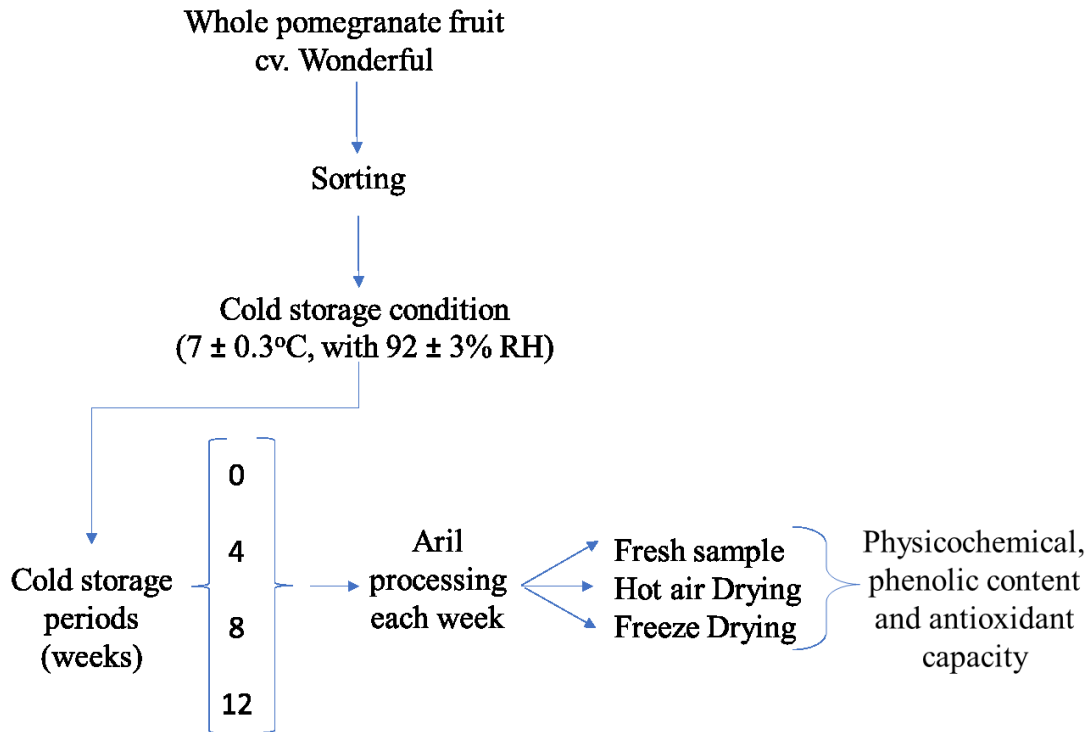
95 % confidence interval. TPC, total phenolic content; TAC, total anthocyanin content; RSA, radical scavenging activity; FRAP, ferric reducing antioxidant power; TCD, total colour difference; TSS, total soluble solids; TA, titratable acidity. ns; non-significant, \* =  $p < 0.05$  and \*\* =  $p < 0.001$  (2-tailed).

**Table 5b**

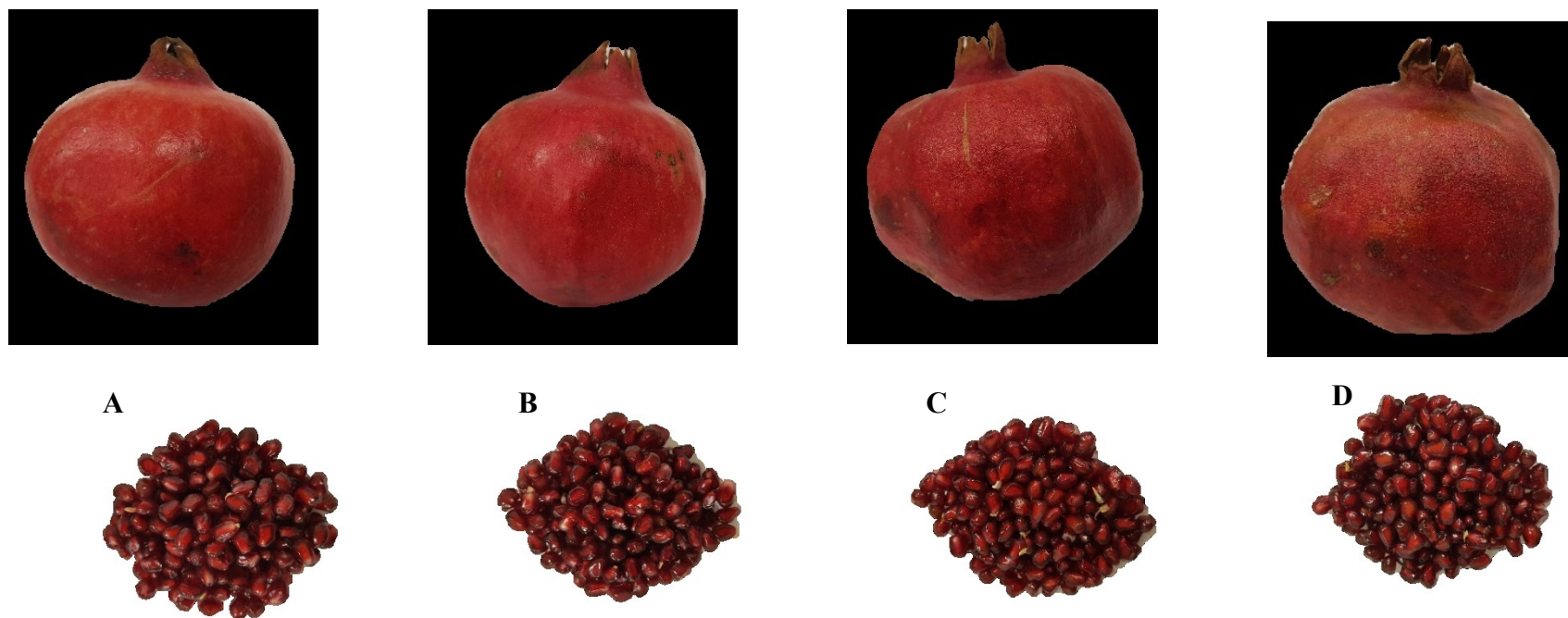
Pearson's correlation coefficients among the investigated parameters of freeze-dried pomegranate arils for 12 weeks storage period

Variables	TCD	TSS	TA	TSS:TA	TPC	TAC	FRAP	RSA
TCD	1							
TSS	-0.685**	1						
TA	-0.138 <sup>ns</sup>	-0.304 <sup>ns</sup>	1					
TSS:TA	-0.590*	0.971**	-0.523*	1				
TPC	0.560*	-0.986**	0.330 <sup>ns</sup>	-0.962**	1			
TAC	0.552 <sup>ns</sup>	-0.983**	0.319 <sup>ns</sup>	-0.956**	-0.947**	1		
FRAP	-0.409 <sup>ns</sup>	0.818**	0.095 <sup>ns</sup>	0.699**	0.997**	-0.878**	1	
RSA	-0.541*	0.969**	-0.228 <sup>ns</sup>	0.920**	0.982**	-0.994**	0.924**	1

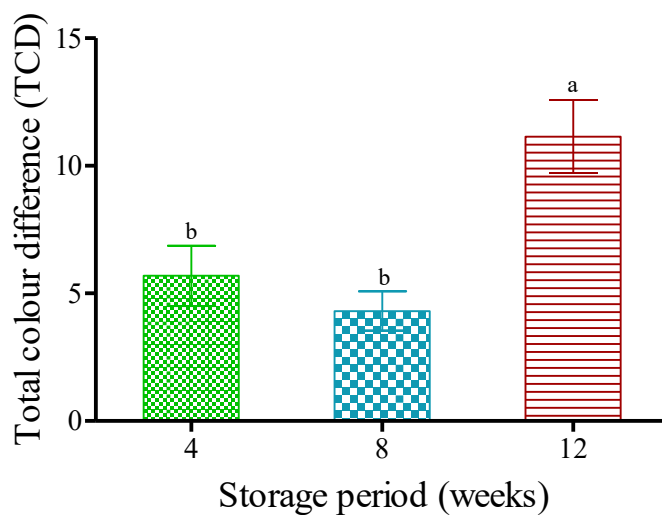
95 % confidence interval. TPC, total phenolic content; TAC, total anthocyanin content; RSA, radical scavenging activity; FRAP, ferric reducing antioxidant power; TCD, total colour difference; TSS, total soluble solids; TA, titratable acidity. ns; non-significant, \* =  $p < 0.05$  and \*\* =  $p < 0.001$  (2-tailed).



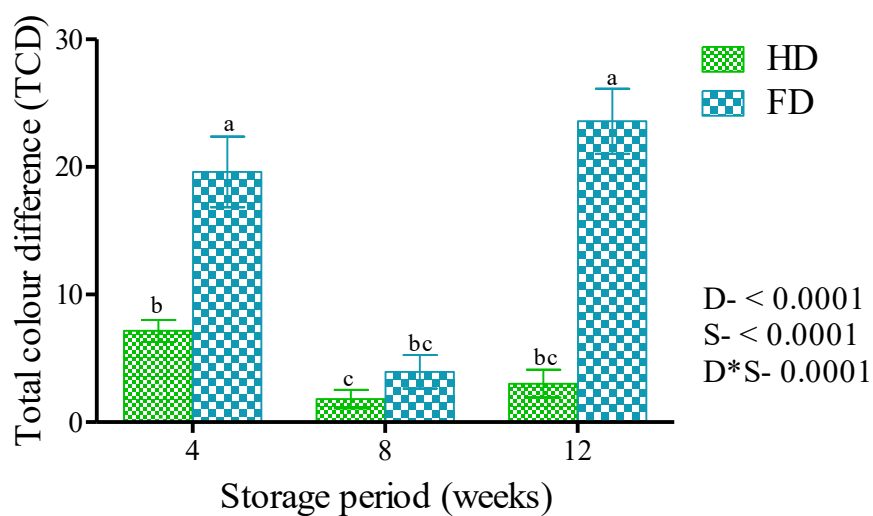
**Fig. 1.** Shows a description of the experimental flowchart



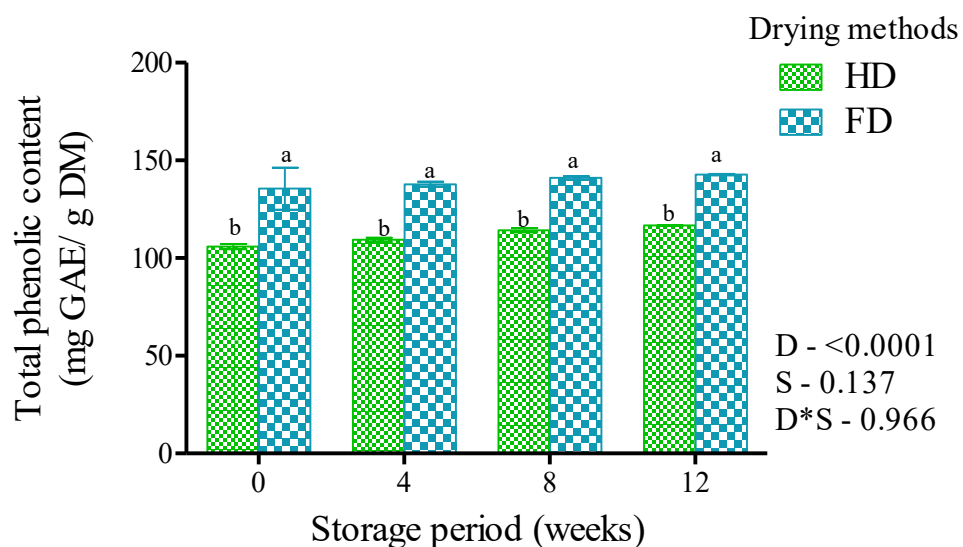
**Fig. 2.** Changes in pomegranate whole fruit (raw material) during cold storage at  $7 \pm 0.3^{\circ}\text{C}$ ,  $92 \pm 3\%$  RH (w.b). (A) at 0 week (B) at 4 weeks (C) at 8 weeks and (D) at 12 weeks storage period. Fresh pomegranate arils showing no noticeable differences visible to the naked eye for the period of 12 weeks.



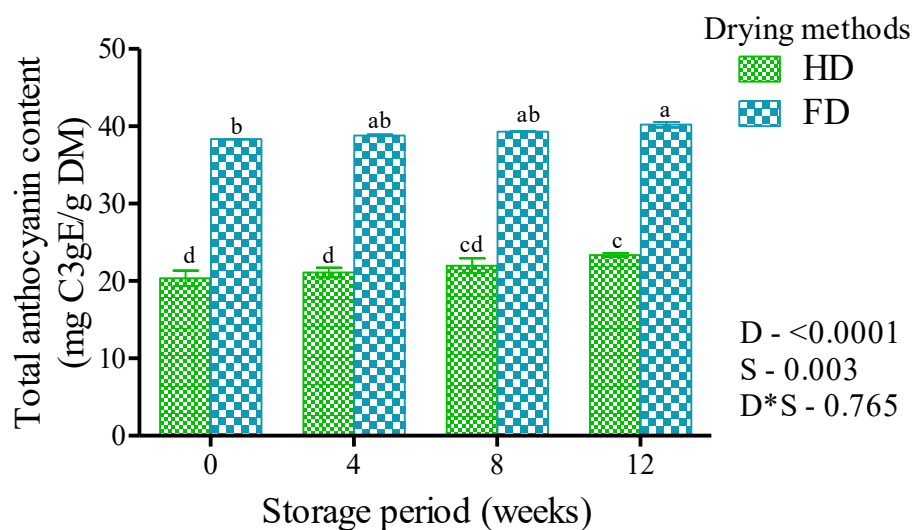
**Fig. 3.** TCD of fresh pomegranate arils during 12 weeks cold storage at  $7 \pm 0.3^{\circ}\text{C}$ ,  $92 \pm 3\%$  RH (w.b). Data presented as means  $\pm$  SE in each row followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.



**Fig. 4.** TCD of dried pomegranate arils during 12 weeks cold storage at  $7 \pm 0.3^{\circ}\text{C}$ ,  $92 \pm 3\%$  RH (w.b). D, drying methods; S, storage period (week). Data presented as means  $\pm$  SE in each row followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.

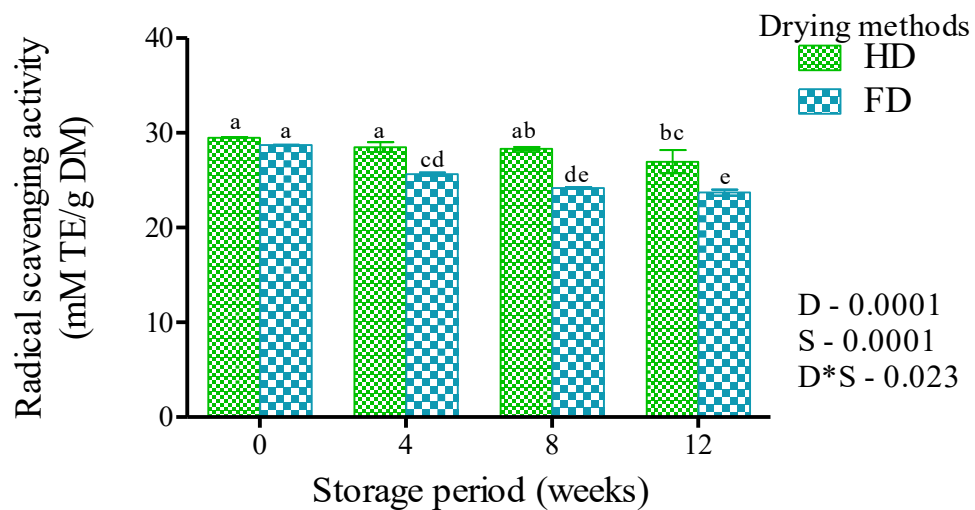


**Fig. 5.** Changes in the total phenolic content of pomegranate dried arils during 12 weeks cold storage at  $7 \pm 0.3^\circ\text{C}$ ,  $92 \pm 3\%$  RH (w.b). HD, hot-air drying; FD, freeze-drying. D, drying methods; S, storage period (week).

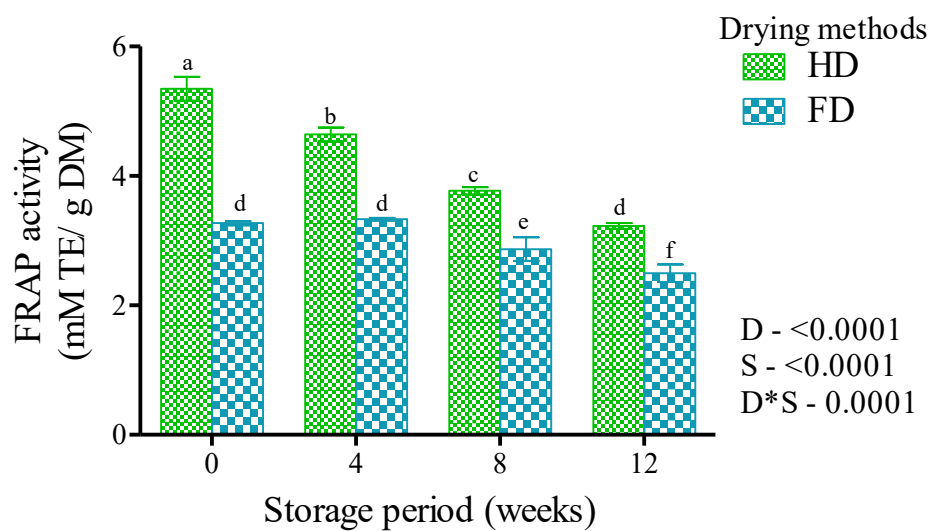


**Fig. 6.** Changes in the total anthocyanin content of pomegranate dried arils during 12 weeks cold storage at  $7 \pm 0.3^\circ\text{C}$ ,  $92 \pm 3\%$  RH (w.b). HD, hot-air drying; FD, freeze-drying. D, drying methods; S, storage period (week).

(a)



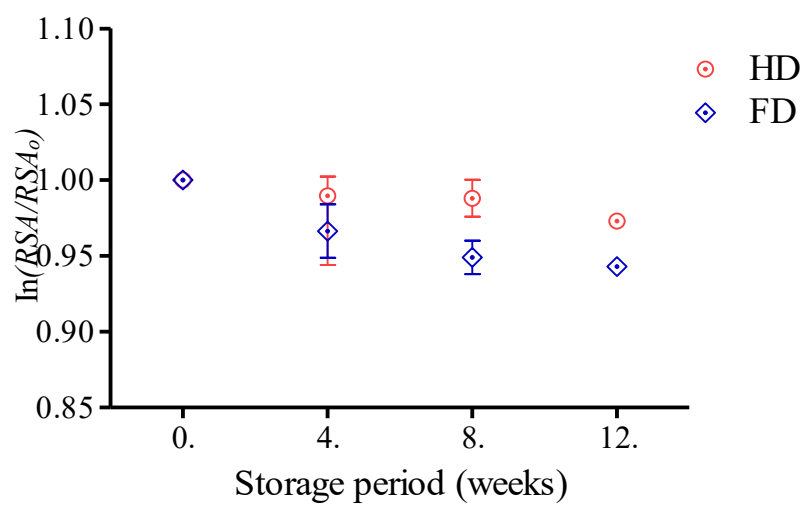
(b)



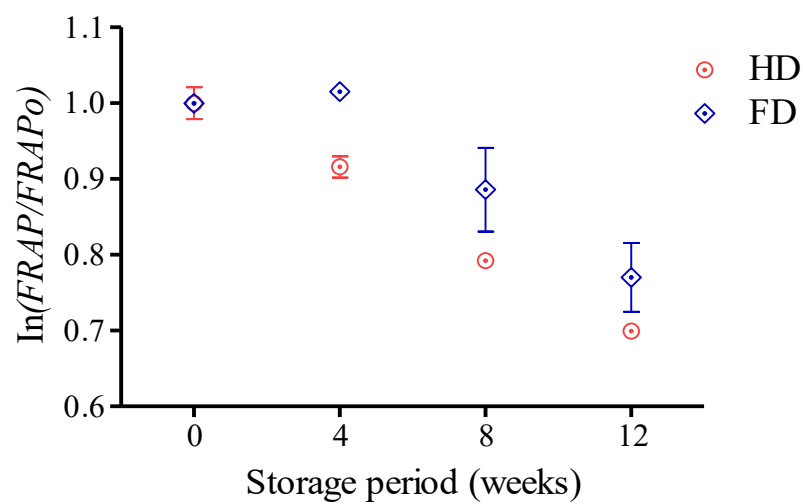
**Fig. 7.** Changes in the antioxidant capacity (a) RSA and (b) FRAP activity of pomegranate dried arils during 12 weeks cold storage at  $7 \pm 0.3^\circ\text{C}$ ,  $92 \pm 3\%$  RH (w.b). HD, hot-air drying; FD, freeze-drying. D, drying methods; S, storage period (week).



(a)



(b)



**Fig. 8.** Degradation kinetics of antioxidant capacity (a) RSA and (b) FRAP of hot-air and freeze-dried pomegranate arils for 12 weeks storage period. HD, hot-air drying; FD, freeze-drying.

## THEME C

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### **Process optimization of blanch-assisted dried arils and powder**

- Mathematical modeling of blanch-assisted dried pomegranate arils in a hot-air dryer (Paper 4)
  - Investigating the effect of blanching on drying kinetics, enzyme inactivation and quality attributes of hot-air dried pomegranate arils (Paper 5)
-

## PAPER 4

### Mathematical modeling of blanch-assisted dried pomegranate (*Punica granatum*) arils in a hot-air drier

**Abstract:** The effect of blanching condition on the hot-air drying kinetics of three pomegranate cultivars ('Acco', 'Herskawitz' and 'Wonderful') were assessed. Water blanching conditions considered were 90°C 30s, 90°C 60s, 100°C 30s and 100°C 60s. The drying experiments were carried out at 60 °C, 19.6 % relative humidity and a constant air velocity of 1.0 m s<sup>-1</sup>. The experimental curves were fitted to seven different drying models. For Acco cultivar, the drying behaviour was best predicted by the Logarithmic and Page model for blanched (R<sup>2</sup> ranging between 0.9966 and 0.9989) and unblanched (R<sup>2</sup> = 0.9918) samples, respectively. Furthermore, for Herskawitz cultivar, Logarithm, Page and Midili models were most suitable to predict drying behaviour for both blanched and unblanched samples. Also, for Wonderful cultivar, Logarithm and Midili models were most accurate to predict the drying behaviour for both blanched and unblanched samples amongst other models. The blanched samples dried faster with a shorter drying time. For instance, 'Acco' (7 h); 'Herskawitz' (8 h); and 'Wonderful' (7 h) than the unblanched samples 15, 20 and 11 h, respectively. Effective diffusion coefficient of moisture in pomegranate arils ranged from 4.81 x 10<sup>-9</sup> and 1.11 x 10<sup>-8</sup> m<sup>2</sup> s<sup>-1</sup> for Acco cultivar, for Herskawitz cultivar; 3.29 x 10<sup>-9</sup> and 1.01 x 10<sup>-8</sup> m<sup>2</sup> s<sup>-1</sup> and for Wonderful cultivar; 5.83 x 10<sup>-9</sup> and 1.09 x 10<sup>-8</sup> m<sup>2</sup> s<sup>-1</sup>. Overall, blanching resulted in low energy consumption during drying of pomegranate arils. In addition, Logarithmic model generally showed an appropriate model for blanched samples regardless of cultivar. For unblanched samples, Page model was more appropriate for 'Acco' and 'Herskawitz' while Midili model was appropriate for 'Wonderful'. This study, therefore, provided science-based and practical drying conditions for the investigated pomegranate cultivars.

**Keywords:** Cultivars, drying kinetics, blanching, effective diffusivity, empirical models

#### 1. Introduction

Pomegranate (*Punica granatum*) fruit consumption has continued to gain global interest among consumers due to its rich nutritional properties and high content of polyphenols (Caleb et al., 2012; Fawole and Opara 2013). It is a good source of phenolic compounds, including flavonoids (anthocyanins, flavonols), condensed tannins (proanthocyanadins) and

hydrolysable tannins (ellagitannins and gallotannins) (Mphahlele et al., 2014). However, pomegranate arils are highly perishable, with shelf-life between 5-8 days (Caleb et al., 2013). This limits pomegranate consumption and availability during the off season. Drying is one of the oldest methods used to preserve food commodities (Ashtiani et al., 2017). Drying has shown to preserve and add value to pomegranate by transforming fresh arils into a nutrient-dense snack called anardana, with improved sensory attributes (Sharma et al., 2016). It has a long shelf-life in proper packages and substantially reduce fruit weight during transportation, lowers handling and storage costs compared to fresh fruit (Ertekin et al., 2010).

Drying of food materials involves the simultaneous heat and moisture transfers between the surface of the material and the surrounding media (Minaei et al., 2012a). In order to preserve food quality during drying, various drying methods have been developed. For instance, in India, the arils are dried in the sun for 10 to 15 days and then sold as a spice (Doymaz, 2012). Calín-Sánchez et al. (2013) investigated the use of freeze, convective and vacuum-microwave dryers for the production of dried pomegranate arils and rind. The authors noted that significant reductions in the contents of sugars, organic acids, and total polyphenols associated with the drying process. Furthermore, Minaei et al. (2012b) compared a thin layer drying behaviour of sour pomegranate arils using the microwave, vacuum, and infrared methods as well as convection drying. The authors reported that microwave pretreatment combined with convective drying performed best for drying of pomegranate arils, taking into consideration the drying rate, effective moisture diffusion and activation energy.

However, several changes may occur in the food due to complex thermo-physical and biochemical process that accompanies the drying process. These changes affect the quality attributes of the end product (Cruz et al., 2014). Reports have shown that high temperature and long drying time affect food physical structures and chemical composition (Vega-Gálvez et al., 2012). These changes include browning, shrinkage or loss of texture and decrease in the bioactive components of the product. For example, according to Krokida et al. (2001), long drying times resulted in significant structural and colour changes in dehydrated apple, banana, carrot and potato. Further, Aguilera et al. (2003) reported that the loss of nutrients during extensive food drying is inevitable. However, these changes could be reduced through pre-treating food materials before drying. A type of pretreatments applied to food materials depends on the type of food to be dried, and the availability of pretreatment (Doymaz, 2010). Several pretreatment methods applied to food materials have been studied to reduce loss of colour due to enzyme inactivation, minimizing drying time and maintaining optimum

nutritional qualities in the food samples (Kingsly et al., 2007). Pretreatment of food materials can be mechanical, such as soaking or dipping food materials in honey, heat either through steam or water blanching and chemical applications using treatment of materials with chemicals such as sulphur, ascorbic acid, sodium metabisulfate (Kingsly et al., 2007; Doymaz, 2010; Adetoro et al., 2018). However, non-chemical pretreatment (mechanical and heat) methods are preferable during food processing due to interference with sensory attributes of pre-treated products (Sabiret et al., 2011), elimination of off-flavours that may have been formed during postharvest, and removal of any residual pesticides (Préstamo et al. 1998; Neves et al., 2012). Studies have shown blanching as a pretreatment method which reduces drying time and preserves quality attributes of food products (Maghoumi et al., 2013).

The analysis of the drying processes assists in validating appropriate operating conditions. Hence, understanding the temperature and moisture behavioural characteristics for the product is a necessity, especially for handling practices and quality control. Kinetic modelling is a useful approach to design and optimize thermal processes in order to maximise quality (Deylami et al., 2016). Several mathematical models have been employed for the purpose of the moisture diffusion process in food products. Moisture diffusivity in solid food can be obtained in different forms which change in accordance with the geometry of the sample and the experimental conditions. These methods have been used to estimate moisture diffusivity and rely on drying kinetics, sorption, or desorption kinetics, as well as moisture profile analysis (Mwithiga et al., 2005).

The mass transfer coefficient of food materials during drying was determined by Dincer and Hussain (2004) to develop new Biot number and lag factor correlation. Mathematical models for pomegranate arils using vacuum and microwave dryer was studied by Minaei et al. (2012) and reported the validity of Midili and Page models. According to Lee and Kim (2009) who reported a tremendous change in effective diffusivity by studying the effect of drying temperature and material thickness of Asian white radish slices. Furthermore, by studying the drying kinetics of pumpkin at different pressure and temperature levels, Arevalo-Pinedo and Fernando (2007) reported that generated values of effective diffusivity for treated samples were smaller than calculated values. Similarly, by investigating the drying model for apples, Goyal et al. (2008) reported that the Logarithmic model best described the drying kinetics in comparison to other models investigated.

Several types of hot-air drying have been implemented to dry crops, the design and operation of such dryers have been a challenge due to the complexity of the parameters that govern the drying process and the factors affecting the quality of the product. One of the most important factors to be measured in dryer design is the drying rate for the prediction of drying time. Hence, investigating the appropriate model suitable for the modelling and prediction of the drying process is crucial. Since cultivar and nature of pretreatment may affect the drying process, it is necessary to establish an appropriate drying model as a function of cultivar and pretreatment as the information remains vital for equipment as well as process design, quality control, and choice of appropriate storage and handling. Therefore, the aims of this study were two-fold, first, to investigate the effect of different blanching conditions on thin-layer drying kinetics of three pomegranate cultivars, and second, to determine the mathematical model that best describe the characteristics of the drying process. In addition, effective diffusivity coefficients for the investigated cultivars were determined.

## **2. Materials and Methods**

### **2.1. Plant material and sample processing**

Three pomegranate cultivars, ‘Acco’, ‘Herskawitz’ and ‘Wonderful’, classified as sweet, sour and sweet-sour, respectively, were investigated. Fruit were harvested at commercial harvest (13°Brix) between February and April in 2018 harvest season from a local orchard in Wellington, South Africa (33°01’00” S, 18°58’59” E). Fruit were sorted for uniformity in size, shape, and colour and transported in an air-conditioned vehicle to the Postharvest Technology Laboratory at Stellenbosch University. The arils were manually separated from the fruits and used for processing. Immediately before the blanching process, 10 g of pomegranate arils per cultivar was taken for initial moisture content determination (Doymaz 2012; Karaaslan et al., 2014). The initial moisture content of the fresh arils was: 56.67, 54.95 and 78.51 % (w.b) for ‘Acco’, ‘Herskawitz’ and ‘Wonderful’, respectively.

### **2.2. Oven drying procedure**

Blanching of fresh arils was carried out in a water bath in batches. Samples were blanched at 90°C and 100°C, each for 30 s and 60 s. The unblanched sample was used as control. Blanched arils were dipped in iced water 0°C for 3 min to halt the continuous heating process and carefully drained before weighing. The process was carried out in triplicates for each treatment. Samples (60 g) was weighed before subjected to drying in an oven (Model nr. 072160, Prolab Instruments, Sep Sci., South Africa) set at a temperature of 60 °C, 19.6 %

relative humidity and  $1.0 \text{ m s}^{-1}$  constant air velocity. The ambient air was used at  $20 \pm 0.3 \text{ }^{\circ}\text{C}$  and 80 - 88 % RH. Weight loss was recorded every 60 min until the desired moisture content between  $10 \pm 0.2 \%$ , wet basis (w.b.) was reached (Minaei et al., 2012a; Doymaz, 2012). A schematic view of a laboratory hot-air convective drying system is illustrated in Figure 1.

### 2.3.Mathematical modeling

To find the most suitable model, seven models) were examined (Table 1. The selected mathematical models best describe drying mechanisms of food material and provide the required temperature and moisture information for proper control of the process (Minaei et al., 2012a). In these models, the moisture ratio was simplified to Eq. (1), according to Doymaz et al. (2016).

$$MR = \frac{M_t}{M_0} \quad (1)$$

Where  $M_t$  is the instantaneous moisture content at time  $t$  (h), and  $M_0$  is the initial moisture content.

### 2.4.Drying rate (DR)

The drying rate of pomegranate arils at a particular time was calculated by Sarpong et al. (2018) in Eq. (2).

$$DR = \frac{M_{t1} - M_{t2}}{t_2 - t_1} \quad (2)$$

Where  $t_1$  and  $t_2$  are the drying times (min) at different times during drying;  $M_{t1}$  and  $M_{t2}$  are the moisture content of samples ( $\text{g min}^{-1}$ ).

### 2.5.Effective moisture diffusivity determination

Fick's second law of diffusion is used to describe the drying process usually controlled by internal diffusion for most biological materials during the falling rate period (Sarpong et al., 2018) and shown in Eq. (3)

$$\frac{\delta M}{\delta t} = D_{eff} \nabla^2 M \quad (3)$$

The effective diffusivity ( $D_{eff}$ ) ( $\text{m}^2 \text{ s}^{-1}$ ) was calculated from diffusion equation (Eq. 3) for the geometry on the assumption of unstable moisture diffusivity, spherical coordinate movement

of moisture, constant temperature and diffusion coefficients and negligible shrinkage during the process of drying is given as the following (Movagharnejad, 2007).

$$MR = \frac{M_t}{M_0} = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp \left[ -\frac{n^2 \pi^2 D_{eff} t}{R^2} \right] \quad (4)$$

$D_{eff}$  is the effective moisture diffusivity ( $m^2 s^{-1}$ ),  $t$  is the time (h),  $R$  denotes the radius of the aril, assumed spherical and constant during the drying period, and  $n$  is a positive integer. In the case of longer drying periods, the above equation can be simplified to the only first term of series, without much affecting the accuracy of the prediction (Minaei et al., 2012; Movagharnejad and Nikzad, 2007; Lopez et al., 2000):

$$\ln(MR) = \ln \frac{6}{\pi^2} - \left( -\frac{\pi^2 D_{eff} t}{R^2} \right) \quad (5)$$

From Eq. (5), a plot of  $\ln MR$  versus drying time gives a straight line with a slope ( $K$ ) of

$$K = \frac{\pi^2 D_{eff}}{R^2} \quad (6)$$

## 2.6. Statistical analysis of the models

Data was processed with STATISTICA (Statistica 13.0, StatSoft Inc., Tulsa, OK, USA) and presented as means  $\pm$  standard error. All analysis was done in triplicates. Factorial analysis of variance (ANOVA) was done in order to observe if the mean values were statistically different and Fisher's LSD test at a level of 95% confidence interval. The moisture ratio curves obtained were fitted with seven mathematical models in order to describe the drying characteristics of blanched and unblanched pomegranate arils. Multiple regression analysis was performed using MATLAB software. The experimental data was evaluated with the coefficient of determination ( $R^2$ ), and the root means square error (RMSE). The higher the  $R^2$  values, and the lower the RMSE values, the better the model of best fit (Wang et al., 2007).

$$R^2 = \frac{\sum_{i=1}^N (MR_i - MR_{pre,i}) \cdot \sum_{i=1}^N (MR_i - MR_{exp,i})}{\sqrt{[\sum_{i=1}^N (MR_i - MR_{pre,i})^2] \cdot [\sum_{i=1}^N (MR_i - MR_{exp,i})^2]}} \quad (7)$$



$$RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^N (MR_{pre,i} - MR_{exp,i})^2} \quad (8)$$

$MR_{exp,i}$  is the  $i$ th experimentally determined moisture ratio,  $MR_{pre,i}$  is the  $i$ th predicted moisture ratio value,  $N$  is the number of observations and  $z$ , the number of drying constants.

### 3. Results and discussion

#### 3.1. Effect of blanching conditions on drying kinetics of dried pomegranate aril

##### 3.1.1. Moisture ratio

Figure 2 shows a pictorial representation of the trend of pomegranate arils before and after processing. At the end of drying, blanched samples became stickier while the unblanched appeared easily separated from each other, which informs part of the textural properties during crushing and grinding (Dak et al., 2014). Also, blanched samples showed glistering dark-purple colour as a result of the occurrence of Maillard reactions during drying (Horuz and Maskan, 2015), while unblanched arils appeared pale. Amongst the investigated cultivars, the order of average drying time blanched sample (100°C 60s) and unblanched sample (control) was observed in the order of cultivar ‘Herskawitz’ (600 min) > ‘Acco’ (420 min) > ‘Wonderful’ (300 min) regardless of blanching condition Fig. 3. Moisture ratio (MR) decreased rapidly with drying time for ‘Acco’, ‘Herskawitz’ and ‘Wonderful’ dried pomegranate arils. It was observed that drying curves showed the same trend with differences in blanching conditions. For instance, moisture content decreased, and the desired moisture was reached faster in blanched samples (420 – 480 min) than the unblanched (660 – 1200 min), regardless of blanching condition Fig 4. This amounted to approximately 53, 60 and 36 % reduction in drying time for ‘Acco’, ‘Herskawitz’ and ‘Wonderful’ respectively.

##### 3.1.2. Drying rate

The variation of drying rate with drying time obtained from Eqn. (2) was shown in Fig 5 for ‘Acco’, ‘Herskawitz’ and ‘Wonderful’, respectively. At early stages, the drying rate increased rapidly, reaching a maximum value, then a progressive decrease with drying time was observed. Drying rates for both blanched and unblanched samples followed a pattern of falling rate period, which is considered as a phenomenon of diffusion-control. A similar trend was observed in the studies by Minaei et al. (2012) for pomegranate arils; Wang et al. (2007) and Kaya et al. (2007) in apple. During the falling rate period, the highest drying rate was found in the blanched samples ranging from (0.358 - 0.398 g min<sup>-1</sup>; 0.274 - 0.363 g min<sup>-1</sup> and 0.333

- 0.382 g min<sup>-1</sup>) for ‘Acco’, ‘Herskawitz’ and ‘Wonderful’, respectively, while all unblanched samples had the least values of drying rate within the range of 0.214 g min<sup>-1</sup> to 0.311 g min<sup>-1</sup> for the three cultivars. The rate of drying for all blanched arils was higher than the unblanched pomegranate arils for ‘Acco’, ‘Herskawitz’ and ‘Wonderful’. Also, the differences in drying were highest at the early stage of drying when the greater amount of water in the sample is evaporated, and at the later stage, the differences in the amount of moisture evaporation in the sample are gradually lower (Fig 5). The higher drying rate is ascribed to the microstructural differences between the blanched and unblanched pomegranate arils. Wang et al. (2007) reported a microstructural difference between fresh apple pomace and the pre-treated apple pomace. This is due to the porous structure and shrinkage of arils during blanching as a result of wide spaces between neighbouring cells (Andres et al., 2004; Bilbao et al., 2000).

### 3.2. Moisture diffusion

The isothermal temperature at 60°C was maintained during the drying process. There were variations in  $D_{\text{eff}}$  for all the blanching conditions regardless of the cultivars Table 2.  $D_{\text{eff}}$  values were within the range of  $4.81 \times 10^{-9}$  and  $1.11 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$  for Acco cultivar, for Herskawitz cultivar;  $3.29 \times 10^{-9}$  and  $1.01 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$  and for Wonderful cultivar;  $5.83 \times 10^{-9}$  and  $1.09 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$ . The highest  $D_{\text{eff}}$  was recorded for samples blanched at 90°C 60s for Acco and Herskawitz cultivars, while (100°C 60s) for Wonderful cultivar. However, control samples for all cultivars had the lowest  $D_{\text{eff}}$  values.

As observed in Figure 4, blanching condition lowers drying time. These results suggested the positive impact of blanching by lowering the drying time of pomegranate arils, regardless of the cultivar. The result from this study corresponds with findings by Karaaslan et al. (2014), who reported the shortest drying time for pre-treated samples of pomegranate aril under vacuum dryer. Sarpong et al. (2018) also reported a 20% reduction in drying time for banana under a relative humidity-convective air dryer. Variability in blanching conditions with similar drying curve indicated that increased temperature or duration during blanching had no distinct differences on the drying kinetics of dried pomegranate arils. This result also agreed with the study by Sarpong et al. (2018), who reported no significant effect on the drying kinetics of dried banana as a result of similar drying curves for all treatments.

Moisture diffusion and drying rate phenomena are dependent on temperature and product composition (Minaei et al., 2012). It is thus logical to attribute the observed differences in moisture diffusion and drying time to initial moisture content in the investigated Acco (56.67

%), Herskawitz (54.95 %) and Wonderful (78.51 %) pomegranate cultivars. Generally, higher values were found in the blanched samples, which is connected to the rapid removal of moisture and faster drying of the samples.  $D_{\text{eff}}$  values reported in this study were close to the range previously reported for pomegranate arils, with  $D_{\text{eff}}$  values of  $0.74 \times 10^{-10}$  to  $5.25 \times 10^{-10} \text{ m}^2 \text{ s}^{-1}$  and  $3.05 \times 10^{-10}$  to  $3.43 \times 10^{-10} \text{ m}^2 \text{ s}^{-1}$  for vacuum and microwave dried arils, respectively (Minaei et al., 2012). High  $D_{\text{eff}}$  values among the blanching conditions indicated that moisture movement in pomegranate arils was in liquid form, a notion reported by Sarpong et al. (2018) for banana slices.

### 3.3. Fitting of drying curve

The  $R^2$  and RMSE were used to determine the goodness of fit model, as shown in Tables 3, 4 and 5. For Acco cultivar, the Logarithmic model gave the highest  $R^2$  value for all blanched samples, which varied from 0.9966 to 0.9989 while Page model, for unblanched samples (0.9918) Table 3. Furthermore, the values of RMSE for the Logarithmic model in Acco cultivar varied from 0.000686 to 0.002839 for blanched samples, while 0.005697 for unblanched samples Table 3. For Herskawitz cultivar, the highest  $R^2$  value was observed in Logarithmic, Page and Midili models for blanched samples varying from 0.9932 to 0.9973 while Midili model had the highest  $R^2$  value for unblanched samples (0.9844) Table 4. Also, the values of RMSE for the Logarithmic, Page and Midili models varied from 0.003201 to 0.004340 for blanched samples, while 0.018195 for unblanched samples Table 4. Similarly, for Wonderful cultivar, the highest  $R^2$  values were observed in Logarithmic and Midili models for blanched samples which varied from 0.9972 to 0.9988 whereas, for unblanched samples, Midili model had the highest  $R^2$  value (0.9929) Table 5. Consequently, the RMSE values for Logarithmic and Midili models for blanched samples varied from 0.001128 to 0.002370 for blanched samples while 0.006619 for unblanched samples Table 5.

From the tables, it was observed that Logarithmic, Page and Midili amongst other models considered for best fit represented the drying characteristics of both blanched and unblanched pomegranate arils for each analysis based on blanching condition and cultivar. The suitability of Logarithmic, Page and Midili models described the observed conformity between  $R^2$  and RMSE values according to Wang et al. (2007), the higher the  $R^2$  values and the lower the RMSE values, the better the model of best fit. This is also similar to the observations of Tunde-Akintunde and Ogunlakin (2013) for drying of pre-treated and untreated pumpkin.

## Conclusion

The effect of blanching on the drying of ‘Acco’, ‘Herskawitz’ and ‘Wonderful’ pomegranate arils in a hot-air dryer was investigated. The blanched samples dried faster with shorter drying time, ‘Acco’ (7 h); ‘Herskawitz’ (8 h); and ‘Wonderful’ (7 h), than the unblanched samples 15, 20 and 11 h respectively. The trend of blanching showed a falling rate as observed against time. Blanched samples had higher drying rate than the unblanched samples. The lowest moisture diffusion was obtained as  $4.81 \times 10^{-9}$ ,  $3.29 \times 10^{-9}$  and  $5.83 \times 10^{-9} \text{ m}^2 \text{ s}^{-1}$  for Acco, Herskawitz and Wonderful cultivars, respectively, while the maximum values being  $1.34 \times 10^{-8}$ ,  $1.19 \times 10^{-8}$  and  $1.29 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$  in the same order of cultivar. The suitability of seven mathematical models to describe the drying behaviour of pomegranate arils was investigated. The model that had the best fit with the highest values of  $R^2$  and lowest values RMSE in Acco cultivar were Logarithmic model for all blanched samples and Page model for the unblanched sample. In Herskawitz cultivar, Logarithmic, Page and Midili models amongst the blanched samples and Page model for unblanched sample while in Wonderful cultivar, Logarithmic and Midili models amongst the blanched samples and Midili model for the unblanched sample. Thus, this model was selected as being suitable to describe the pomegranate aril drying process for the blanching conditions considered.

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**Table 1**

Empirical models describing blanched and unblanched pomegranate aril drying kinetics

Model	Name	Emperical expression	References
1	Lewis	$MR = \exp(-kt)$	Wang et al. (2007)
2	Henderson and Pabis	$MR = a.\exp(-kt)$	Wang et al. (2007)
3	Logarithmic	$MR = a.\exp(-kt) + c$	Yagcioglu et al. (1999)
4	Page	$MR = \exp(-kt^n)$	Diamante et al. (2010)
5	Wang and Singh	$MR = 1 + at + bt^2$	Kaleta and Gornicki (2010)
6	Modified Page	$MR = \exp(-kt)^n$	Wang et al. (2007)
7	Midili	$MR = a.\exp(-kt^n) + bt$	Menges and Ertekin (2005)

**Table 2**Moisture effective diffusion ( $D_{\text{eff}}$ ) of dried pomegranate aril under different blanching conditions for different cultivars

Cultivar/classification	Blanching condition	Moisture diffusivity ( $\text{m}^2/\text{s}$ )
Acco	Control	$4.81 \times 10^{-9}$
	90°C 30s	$1.24 \times 10^{-8}$
	90°C 60s	$1.34 \times 10^{-8}$
	100°C 30s	$1.11 \times 10^{-8}$
	100°C 60s	$1.24 \times 10^{-8}$
Herskawitz	Control	$3.29 \times 10^{-9}$
	90°C 30s	$1.04 \times 10^{-8}$
	90°C 60s	$1.19 \times 10^{-8}$
	100°C 30s	$1.11 \times 10^{-8}$
	100°C 60s	$1.01 \times 10^{-8}$
Wonderful	Control	$5.83 \times 10^{-9}$
	90°C 30s	$1.09 \times 10^{-8}$
	90°C 60s	$1.14 \times 10^{-8}$
	100°C 30s	$1.27 \times 10^{-8}$
	100°C 60s	$1.29 \times 10^{-8}$



**Table 3**

Curve fitting criteria for various mathematical models and parameters for blanched and unblanched pomegranate arils cv. Acco

Model number	$k$	$a$	$b$	$c$	$n$	Determining coefficient ( $R^2$ )	Root mean square error (RMSE)
<b>90°C 30s</b>							
1	$1.01 \times 10^{-4}$					0.9821	0.017932
2	$9.63 \times 10^{-5}$	0.9550				0.9779	0.015464
3	$1.49 \times 10^{-4}$	0.8611		0.1365		0.9989	0.000686
4	$1.02 \times 10^{-3}$				0.7371	0.9949	0.02257
5		$-8.75 \times 10^{-5}$	$2.22 \times 10^{-9}$			0.9804	0.01557
6	$1.08 \times 10^{-5}$				9.3788	0.9821	0.017933
7	$1.59 \times 10^{-3}$	1.01107	$9.30 \times 10^{-7}$		0.7097	0.9949	0.003182
<b>90°C 60s</b>							
1	0.00010					0.9880	0.011722
2	$9.91 \times 10^{-5}$	0.9687				0.9857	0.010517
3	0.000138	0.8899		0.1101		0.9979	0.001314
4	0.00064				0.8036	0.9940	0.003859
5		$-8.75 \times 10^{-5}$	$2.22 \times 10^{-9}$			0.9877	0.010128
6	$1.09 \times 10^{-5}$				9.4298	0.9880	0.011722
7	0.00159	1.0111	$9.3 \times 10^{-7}$		0.7097	0.9905	0.007004
<b>100°C 30s</b>							
1	0.00010					0.9682	0.031770
2	$9.3 \times 10^{-5}$	0.9550				0.9628	0.027411
3	0.00015	0.8611		0.1365		0.9966	0.002839
4	0.00118				0.7371	0.9914	0.006068
5		$-8.75 \times 10^{-5}$	$2.22 \times 10^{-9}$			0.9691	0.024845
6	$1.09 \times 10^{-5}$				9.4298	0.9694	0.032035
7	0.00159	1.0111	$9.3 \times 10^{-7}$		0.7097	0.9947	0.003418
<b>100°C 60s</b>							
1	0.00010					0.9753	0.022735
2	$9.9 \times 10^{-5}$	0.8987				0.9721	0.023406
3	0.00015	0.8611		0.1365		0.9977	0.002243
4	0.00118				0.7371	0.9938	0.004394
5		$-8.8 \times 10^{-5}$	$2.22 \times 10^{-9}$			0.9749	0.021579
6	$1.08 \times 10^{-5}$				9.9287	0.9794	0.022169
7	0.00159	1.0111	$9.30 \times 10^{-7}$		0.7097	0.9958	0.003968
<b>Control</b>							
1	$3.97 \times 10^{-5}$					0.9525	0.094019
2	$3.22 \times 10^{-5}$	0.8519				0.9353	0.047809
3	$2.68 \times 10^{-5}$	0.7439		0.2561		0.9871	0.009322
4	0.00249				0.5933	0.9918	0.005697
5		$-3.50 \times 10^{-5}$	$3.90 \times 10^{-10}$			0.9599	0.075367
6	$9.98 \times 10^{-6}$				3.99	0.9527	0.094028
7	0.00185	0.99412	$4.33 \times 10^{-7}$		0.6254	0.9920	0.005833

1; Lewis, 2; Henderson and Pabis, 3; Logarithmic, 4; Page, 5; Wang and Singh, 6; Modified Page, 7; Midili.

**Table 4**

Curve fitting criteria for various mathematical models and parameters for blanched and unblanched pomegranate arils cv. Herskawitz

Model number	$k$	$a$	$b$	$c$	$n$	Determining coefficient ( $R^2$ )	Root mean square error (RMSE)
<b>90°C 30s</b>							
1	$7.61 \times 10^{-5}$					0.9821	0.017932
2	$7.27 \times 10^{-5}$	0.9611				0.9779	0.015464
3	0.00011	0.8611		0.1365		0.9989	0.000686
4	0.001				0.7371	0.9949	0.02257
5		$-8.75 \times 10^{-5}$	$2.22 \times 10^{-9}$			0.9804	0.01557
6	$9.17 \times 10^{-6}$				8.2998	0.9821	0.017933
7	0.00129	1.0211	$9.30 \times 10^{-7}$		0.7097	0.9949	0.003182
<b>90°C 60s</b>							
1	$6.42 \times 10^{-5}$					0.9880	0.011722
2	$6.33 \times 10^{-5}$	0.9750				0.9857	0.010517
3	0.000109	0.8711		0.1765		0.9979	0.001314
4	0.00018				0.8916	0.9940	0.003859
5		$-8.35 \times 10^{-5}$	$2.22 \times 10^{-9}$			0.9877	0.010128
6	$8.43 \times 10^{-6}$				7.6155	0.9880	0.011722
7	0.00109	1.0111	$9.2 \times 10^{-7}$		0.7097	0.9905	0.007004
<b>100°C 30s</b>							
1	$7.63 \times 10^{-5}$					0.9682	0.031770
2	$7.33 \times 10^{-5}$	0.9550				0.9628	0.027411
3	0.00011	0.8011		0.1565		0.9966	0.002839
4	0.0002				0.8991	0.9914	0.006068
5		$-8.40 \times 10^{-5}$	$2.22 \times 10^{-9}$			0.9691	0.024845
6	$9.19 \times 10^{-6}$				8.3037	0.9694	0.032035
7	0.00119	1.0011	$9.0 \times 10^{-7}$		0.7097	0.9947	0.003418
<b>100°C 60s</b>							
1	$6.53 \times 10^{-5}$					0.9753	0.022735
2	$6.53 \times 10^{-5}$	0.9650				0.9721	0.023406
3	$6.75 \times 10^{-5}$	0.9482		0.0295		0.9977	0.002243
4	0.00016				0.9048	0.9938	0.004394
5		$-9.0 \times 10^{-5}$	$2.22 \times 10^{-9}$			0.9749	0.021579
6	$7.82 \times 10^{-6}$				8.3567	0.9794	0.022169
7	0.00129	1.0011	$2.21 \times 10^{-6}$		0.7097	0.9958	0.003968
<b>Control</b>							
1	$2.96 \times 10^{-5}$					0.9525	0.094019
2	$3.22 \times 10^{-5}$	0.9819				0.9353	0.047809
3	$1.29 \times 10^{-4}$	0.9611		0.2665		0.9871	0.009322
4	0.00209				0.5933	0.9918	0.005697
5		$-3.50 \times 10^{-5}$	$3.90 \times 10^{-10}$			0.9599	0.075367
6	$9.98 \times 10^{-6}$				2.99	0.9527	0.094028
7	0.00145	0.99412	$5.33 \times 10^{-7}$		0.6254	0.9920	0.005833

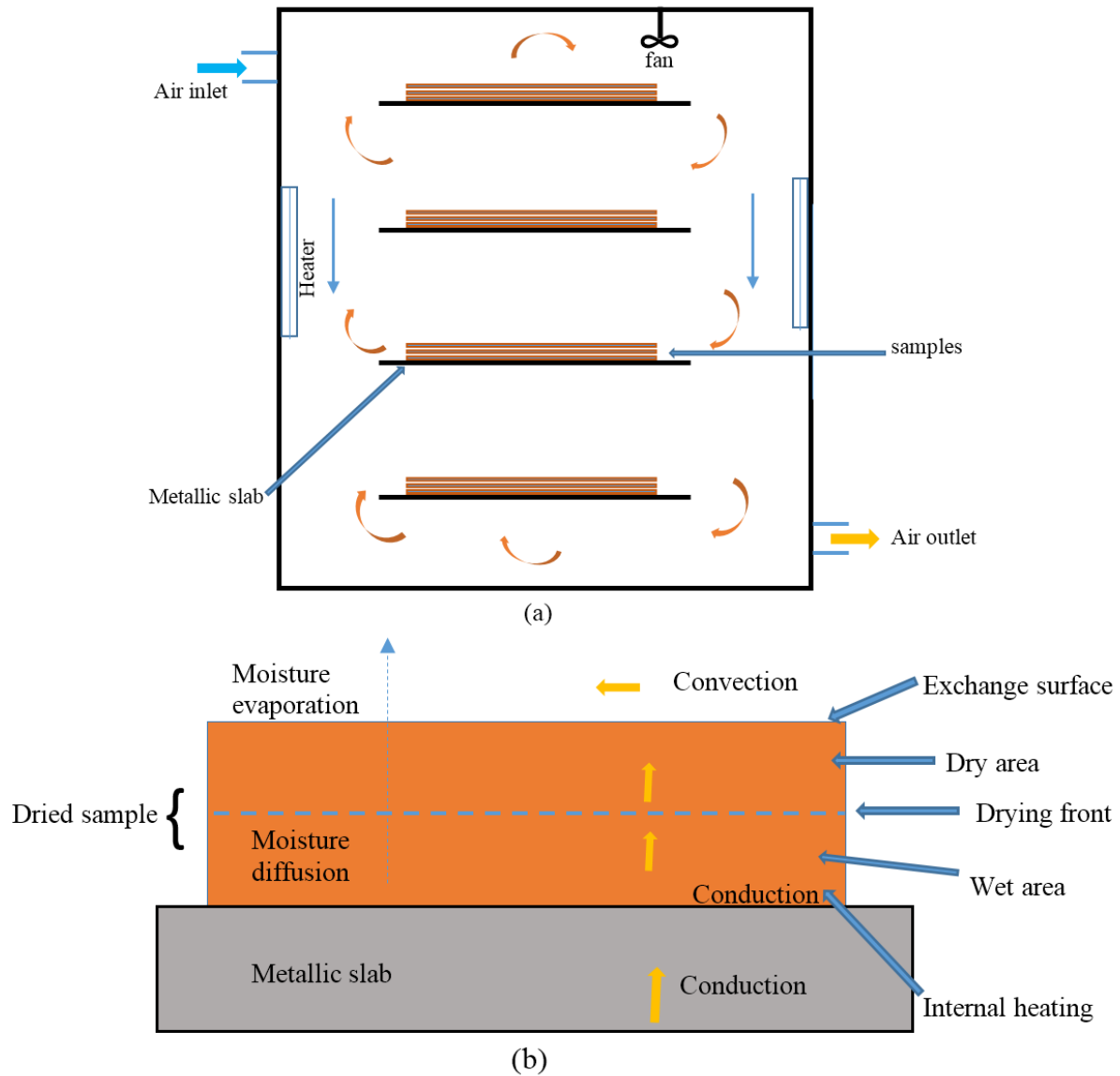
1; Lewis, 2; Henderson and Pabis, 3; Logarithmic, 4; Page, 5; Wang and Singh, 6; Modified Page, 7; Midili.

**Table 5**

Curve fitting criteria for various mathematical models and parameters for blanched and unblanched pomegranate arils cv. Wonderful

Model number	$k$	$a$	$b$	$c$	$n$	Determining coefficient ( $R^2$ )	Root mean square error (RMSE)
<b>90°C 30s</b>							
1	$1.02 \times 10^{-4}$					0.9821	0.017932
2	$9.33 \times 10^{-5}$	0.9880				0.9779	0.015464
3	$1.19 \times 10^{-4}$	0.8611		0.1365		0.9989	0.000686
4	$1.07 \times 10^{-3}$				0.7371	0.9949	0.02257
5		$-8.75 \times 10^{-5}$	$2.22 \times 10^{-9}$			0.9804	0.01557
6	$1.01 \times 10^{-5}$				9.1787	0.9821	0.017933
7	$1.39 \times 10^{-3}$	1.01107	$9.30 \times 10^{-7}$		0.7097	0.9949	0.003182
<b>90°C 60s</b>							
1	$1.01 \times 10^{-4}$					0.9880	0.011722
2	$9.95 \times 10^{-5}$	0.9999				0.9857	0.010517
3	$1.18 \times 10^{-4}$	0.8899		0.1101		0.9979	0.001314
4	$5.2 \times 10^{-4}$				0.8046	0.9940	0.003859
5		$-8.75 \times 10^{-5}$	$2.22 \times 10^{-9}$			0.9877	0.010128
6	$1.00 \times 10^{-5}$				9.0898	0.9880	0.011722
7	0.00139	1.0011	$9.3 \times 10^{-7}$		0.7097	0.9905	0.007004
<b>100°C 30s</b>							
1	0.00010					0.9682	0.031770
2	$9.97 \times 10^{-5}$	0.9850				0.9628	0.027411
3	$1.49 \times 10^{-4}$	0.8611		0.1365		0.9966	0.002839
4	0.00118				0.7371	0.9914	0.006068
5		$-8.75 \times 10^{-5}$	$2.22 \times 10^{-9}$			0.9691	0.024845
6	$1.08 \times 10^{-5}$				9.5787	0.9694	0.032035
7	0.00159	1.0111	$9.3 \times 10^{-7}$		0.7097	0.9947	0.003418
<b>100°C 60s</b>							
1	$1.01 \times 10^{-4}$					0.9753	0.022735
2	$9.96 \times 10^{-5}$	0.9720				0.9721	0.023406
3	$1.49 \times 10^{-4}$	0.8611		0.1265		0.9977	0.002243
4	0.00138				0.7271	0.9938	0.004394
5		$-8.85 \times 10^{-5}$	$2.22 \times 10^{-9}$			0.9749	0.021579
6	$1.08 \times 10^{-5}$				9.7987	0.9794	0.022169
7	0.00169	1.0111	$9.30 \times 10^{-7}$		0.7097	0.9958	0.003968
<b>Control</b>							
1	$4.97 \times 10^{-5}$					0.9525	0.094019
2	$3.52 \times 10^{-5}$	0.7506				0.9353	0.047809
3	$9.99 \times 10^{-5}$	0.7439		0.2561		0.9871	0.009322
4	0.00279				0.5933	0.9918	0.005697
5		$-3.50 \times 10^{-5}$	$3.90 \times 10^{-10}$			0.9599	0.075367
6	$9.98 \times 10^{-6}$				4.99	0.9527	0.094028
7	0.00185	0.94412	$4.33 \times 10^{-7}$		0.6254	0.9920	0.005833

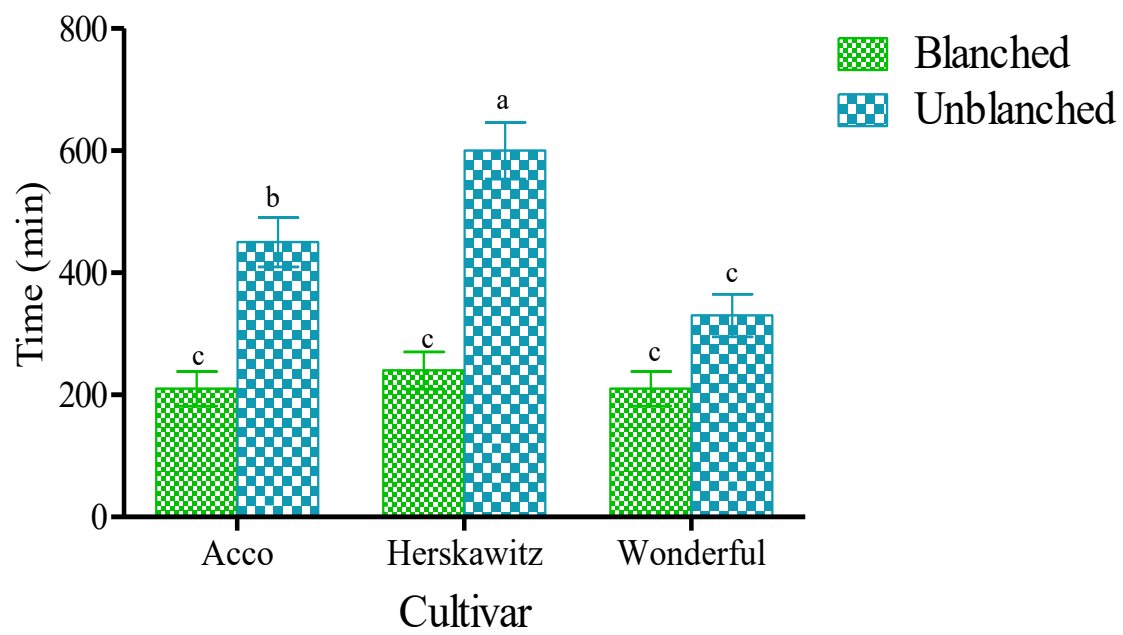
1; Lewis, 2; Henderson and Pabis, 3; Logarithmic, 4; Page, 5; Wang and Singh, 6; Modified Page, 7; Midili.



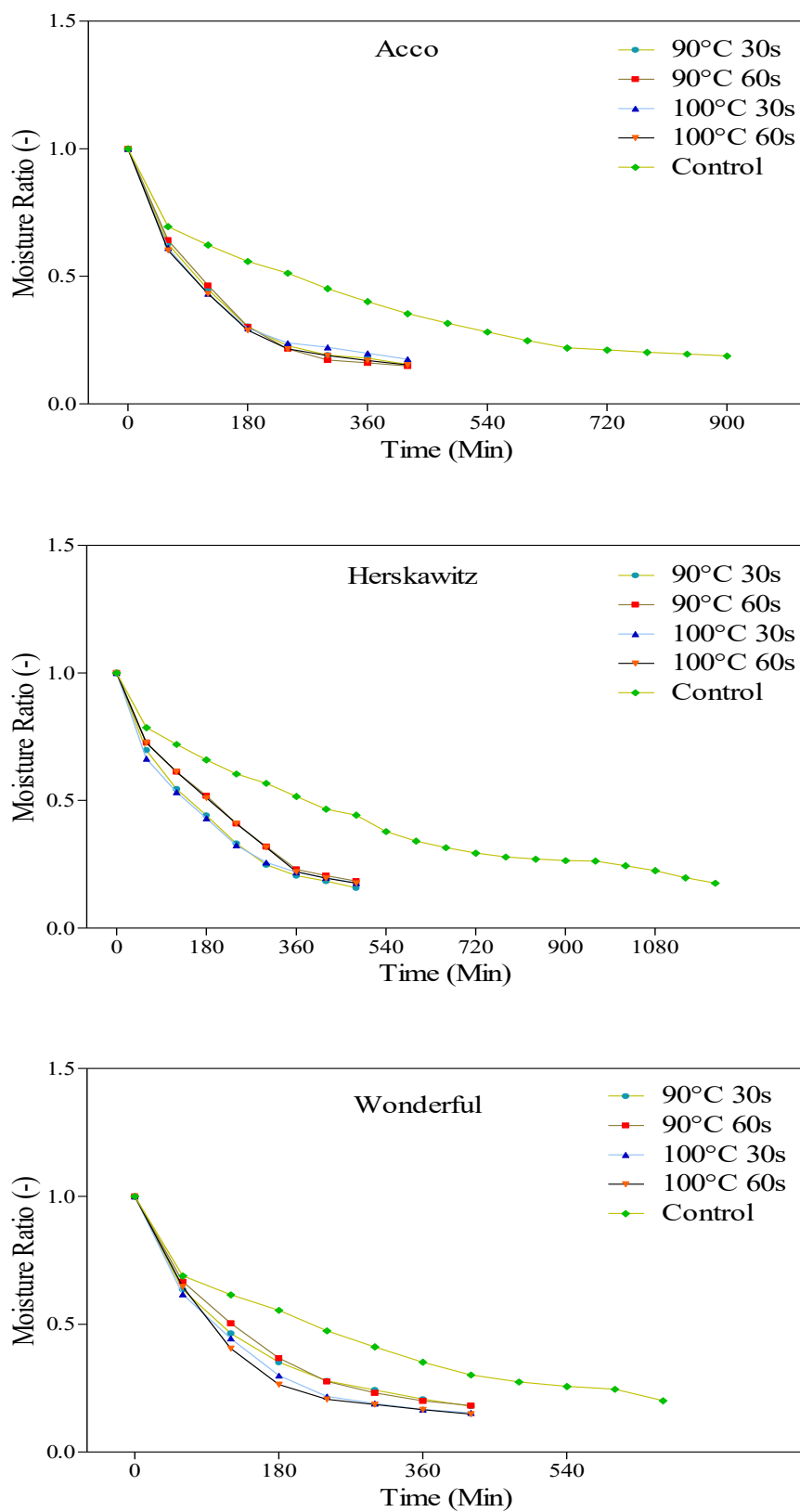
**Fig. 1.** Experimental setup for the drying experiment. (a) Schematic diagram showing the laboratory hot-air convective drying system and (b) demonstrating the mechanism of heat and mass transfer through the sample.



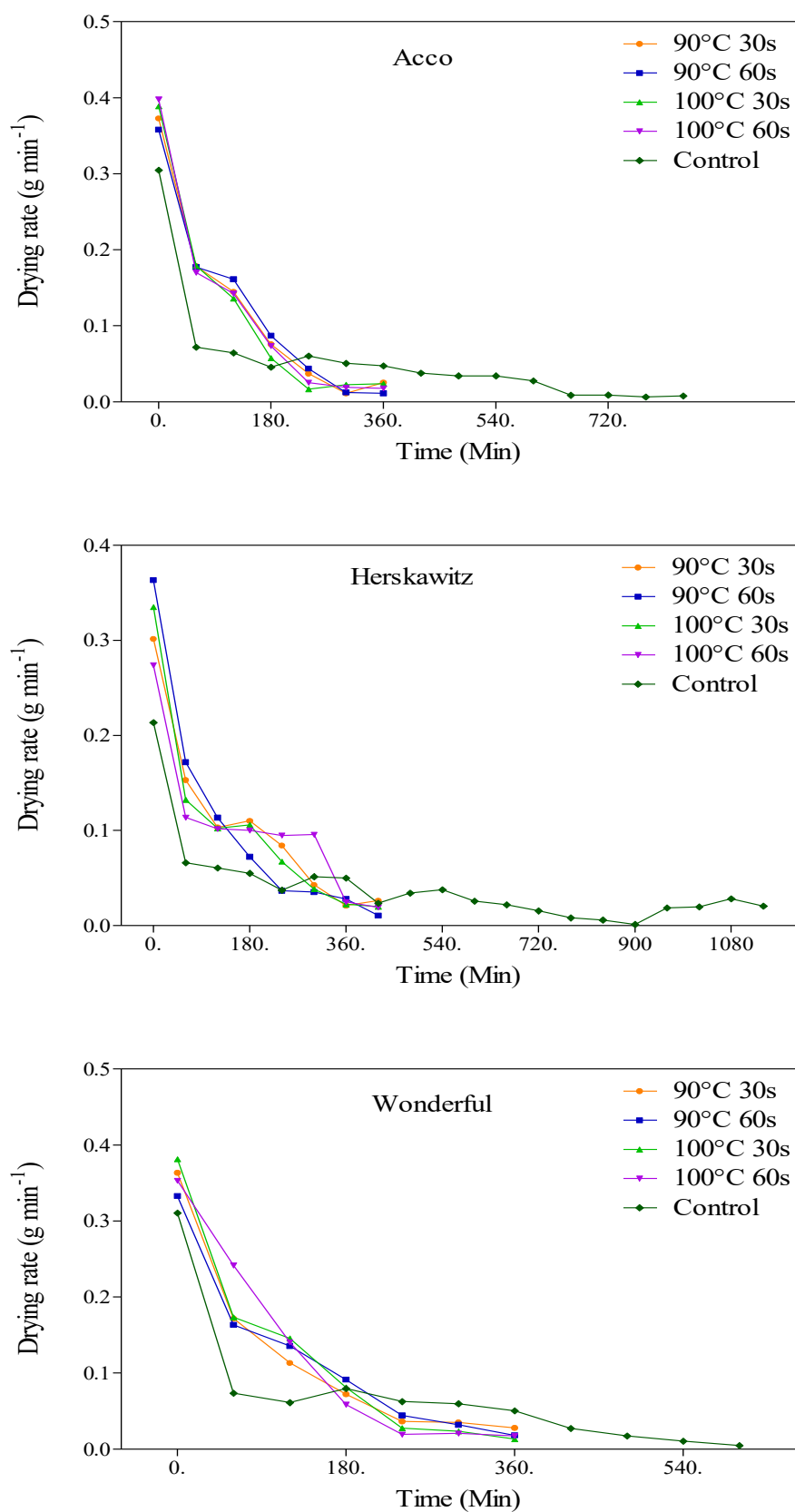
**Fig. 2.** Pomegranate arils at different stages (a) samples before processing (b) blanch-assisted dried arils (c) unblanched dried arils (control).



**Fig. 3.** Graph comparing drying time (min) against cultivars Acco, Herskawitz and Wonderful for samples blanched at 100 °C 60s drying with a hot-air flow rate of 1 m s<sup>-1</sup>, temperature condition 60 °C and 19.6 % RH.



**Fig. 4.** Drying curves for pomegranate aril cultivars Acco, Herskawitz and Wonderful at a hot-air flow rate of  $1 \text{ m s}^{-1}$ , temperature condition  $60^\circ\text{C}$  and  $19.6\%$  RH.



**Fig. 5.** Drying rate versus drying time of pomegranate arils for cultivars Acco, Herskawitz and Wonderful at a hot-air flow rate of 1 m s<sup>-1</sup>, temperature condition 60 °C and 19.6 % RH.



## PAPER 5

# Effect of blanching on drying kinetics, enzyme inactivation and quality attributes of hot-air dried pomegranate (*Punica granatum* L.) arils (cv. Wonderful)

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### ABSTRACT

Blanch-assisted hot-air drying of pomegranate arils with blanching treatments 90°C for 30s, 100°C for 60s and unblanched (control) arils were investigated. Relationships between blanching on moisture ratio, drying rate, enzyme inactivation and other qualities of dried arils were discussed. The drying experiments were carried out at 60 °C, 19.6 % relative humidity and air velocity of 1.0 m s<sup>-1</sup>. Results showed that blanching reduced drying time by approximately 36.4 %, regardless of treatment. Unblanched arils had the least effective moisture diffusivity (5.83 x 10<sup>-9</sup> m<sup>2</sup> s<sup>-1</sup>) compared to blanched arils at 90°C 30s and 100°C 60s with moisture diffusivity of 1.09 x 10<sup>-8</sup> and 1.29 x 10<sup>-8</sup> m<sup>2</sup> s<sup>-1</sup>, respectively. Also, blanching reduced enzyme activity by 76 and 68 % for blanched arils treated at 90°C for 30s and 100°C for 60s, respectively, compared to unblanched arils. For the total colour difference (TCD), unblanched arils were 20.9 and 16.6 % higher than blanched arils treated at 90°C for 30s and 100°C for 60s, respectively. Furthermore, the total soluble solids (TSS) for unblanched aril increased significantly from 16.1 to 24.9 °Brix after drying, followed by arils treated at 90°C for 30s and 100°C for 60s (21.4; 18.5 °Brix), respectively. Among the blanching treatments, dried arils treated at 90°C for 30s had the highest total anthocyanin content (28.6 mg C3gE/ g DM), followed by 100°C for 60s (24.8 mg C3gE/ g DM). Similarly, dried arils treated at 90°C for 30s had the highest radical scavenging activity (RSA) (32.1 mM TE/g DM) while the least was observed with unblanched arils (17.0 mM TE/g DM). Overall, the parameters reflected distinct differences in quality with blanching treatments.

Keywords: Peroxidase, phenolic content, antioxidants, texture, PCA, *Punica granatum* L.

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### 1. Introduction

Pomegranate (*Punica granatum* L.) is a tropical and subtropical fruit grown around the world, including Asia, USA, Russia, North Africa, and Spain (Al-Said et al., 2009; Holland et al., 2009; Fawole et al., 2012). In the past five years, South Africa has increased its commercial production; accounting for approximately 1.4 million cartons exported in 2017 compared to about 400,000 cartons exported in 2012 (POMASA, 2018). Considering the high nutritional and polyphenolic properties in pomegranate, consumers have consciously shifted their interest

towards the fruit due to continuous increase in its consumption (Fawole and Opara 2013a). The fruit comprises of a non-edible (peel) and the edible portion (arils), the arils contains juice and seed/kernel, with the arils arranged in sac-like structure compartments (Caleb et al., 2012; Fawole and Opara 2013a). These have been identified as a potential product of functional food with a high level of biochemical compounds as well as reported health benefits (Espín et al., 2007; Fawole and Opara 2013b; Fawole et al., 2015). Its consumption is eminently taken as juice and is used largely in the industry to manufacture food-related materials such as jellies, jams, flavouring and colouring agents (Opara et al., 2009).

Several metabolic activities occur during fruit development which results in significant changes in the visual and organoleptic properties of the fruit, reducing the marketability of the harvested product (Lara et al., 2014). Further, damages on fruit is popularly the cause for quality deterioration and postharvest losses during handling and storage (Jahanfar et al., 2011; Hussein et al., 2017). Damaging occurrences such as cracks, bruises and black spots in pomegranate fruit results in high postharvest losses to the pomegranate sector. For instance, cracks in the fruit at maturity are a constraint resulting in 20 - 40 % loss in fruits (POMASA 2012) which rarely fetch reasonable prices to growers. Also, pomegranate arils are highly perishable with very short shelf-life (Caleb et al., 2012). This limits its availability in the market during the off season. Drying is a useful means to extend the shelf-life of the fruit and its co-products, such as arils (Ratti 2001). It involves the use of heat to vaporize the water present in the food, as well as protect food from adverse effects of microbial and enzymatic activities (Ratti 2001; Kim et al., 2002; Tang et al., 2013). This enables food products to be stored for more extended periods since the activities of microorganisms and enzymes are inhibited through the drying process (Alibas et al., 2001). Thermal processing affects food-value compounds such as pigments, anthocyanin and phenolic compounds (Fazaeli et al., 2013). More so, fruit-cut before drying also causes discolouration due to browning pigmentation (Adetoro et al., 2017; Thakur et al., 2010). The enzymes responsible for these activities include polyphenol oxidase and peroxidase (Meighani et al., 2014; Arendse et al., 2018). They oxidize mono- and di-phenols to form o-quinones which appears as brown pigments (Sarpong et al., 2018).

Pomegranate arils have a problem of poor quality in part, due to discolouration (enzymatic browning) during handling and storage and as a result, it commands a low premium in the market (Thakur et al., 2010). Due to the arils, high moisture content ranging between 50 to 80 % wet basis depending on the agronomic practices and geographical location, the

extended drying period is often required which often reduces the sensory quality (taste, texture) and antioxidant properties. Therefore, addressing such cases are very crucial to enhance the quality and marketability of the dried product. One of the common ways to reduce the process of enzymatic browning in a food material is by inhibiting the activity of the enzymes through pretreatment (Tembo et al., 2008). Several pretreatment methods can be applied depending on the food material to be dried, its end product, and availability (Doymaz, 2010). Many studies have reported the successful application of pretreatments such as water blanching, steam blanching, sulphiting, citric and ascorbic acid in its application to control enzymatic browning (Thakur et al., 2010; Adetoro et al., 2017; Sarpong et al., 2018). Blanching is one of the pretreatment methods often used to inhibit physiological processes before drying of minimally processed vegetables and fruits. It is a heat pretreatment method which inactivates the enzymatic agent causing the unacceptable discolouration and off-flavours (Doymaz, 2010) and further stabilizes bioactive compounds during processing and storage (Deylami et al., 2016). The process of blanching involves heating the food materials with steam or hot water prior to drying (Maté et al., 1999). Maghoumi et al. (2013) reported the use of hot water dipping on pomegranate arils heated at 55°C for 30s and observed that hot water suppressed the polyphenol oxidase activity. Similarly, Thakur et al. (2010) investigated steam blanching pretreatment to prevent the discolouration of dried arils, and they found out that pre-treated samples had highest scores for sensory characteristics such as colour, texture, taste, aroma and overall acceptability. Blanching also significantly decreased the colour chroma of *Ziziphus mauritiana* fruits in comparison with non-blanching fruits (Tembo et al., 2008).

Wonderful pomegranate cultivar is one of the most popularly consumed and exported pomegranate cultivars in South Africa. In order to reduce enzyme activity and maintain the quality attributes in dried pomegranate arils, this experiment was conducted. Therefore, the objectives of this study were; i) to investigate the effect of blanching treatments on drying characteristics and enzymes inactivation of pomegranate arils; ii) to quantify the impact of blanching on the quality attributes of pomegranate arils cv. Wonderful.

## **2. Materials and Methods**

### **2.1. Fruit material and processing**

Pomegranate fruit (cv. Wonderful) were harvested at commercial maturity from Blydeverwacht orchard, Wellington, South Africa (33°01'00" S, 18°58'59" E). Fruit were sorted for uniformity in size, shape, and colour and transported in an air-conditioned vehicle to

the Postharvest Technology Laboratory at Stellenbosch University. Pomegranate arils were carefully extracted and used for immediate processing. Ten grams of pomegranate arils was taken for initial moisture content determination using a moisture analyzer (KERN DBS 60-3 Balingen, Germany) (Doymaz, 2012). The initial moisture content of the fresh arils was 78.56 % (w.b.).

### 2.1.1. Oven drying procedure

Blanching of fresh arils was carried out in a water bath in batches. Arils were blanched at 90°C for 30s and 100°C for 60s. Unblanched arils were used as the control. Blanched arils were dipped in iced water at 0°C for 3 min to halt the continuous heating process and carefully drained before being weighed. The process was carried out in triplicates for each treatment. Arils (60 g) was weighed before subjected to drying in an oven (Model nr. 072160, Prolab Instruments, Sep Sci., South Africa) set at 60°C, 19.6 % relative humidity (RH) and 1.0 m s<sup>-1</sup> constant air velocity. The ambient air was used at 20 ± 0.3°C and 80 - 88 % RH. From the initial moisture content 78.56 ± 0.03 %, weight loss was recorded hourly until the desired moisture content between 10 - 12 ± 0.2 % wet basis (w.b.) was reached.

### 2.1.2. Drying characteristics

Moisture ratio (MR) for hot-air dried arils was calculated according to Eq. (1) (Doymaz et al., 2016).

$$MR = \frac{M_t}{M_0} \quad (1)$$

where  $M_t$  is the instantaneous moisture content at time  $t$  (h), and  $M_0$  is the initial moisture content.

The drying rate (DR) of pomegranate arils at a particular time was calculated by Adetoro et al. (2020) in Eq. (2).

$$DR = \frac{M_{t1} - M_{t2}}{t_2 - t_1} \quad (2)$$

where  $t_1$  and  $t_2$  are the drying times (min) at different times during drying;  $M_{t1}$  and  $M_{t2}$  are the moisture content of arils (g min<sup>-1</sup>).

### 2.1.3. Effective moisture diffusivity

Fick's second law of diffusion was used to describe drying process usually controlled by internal diffusion for most biological materials during the falling rate period (Sarpong et al., 2018) and shown in Eq. (3)

$$\frac{\delta M}{\delta t} = D_{eff} \nabla^2 M \quad (3)$$

The effective diffusivity ( $D_{eff}$ ) ( $m^2 s^{-1}$ ) was calculated from diffusion equation (Eq. 3) for the geometry on assumption of unstable moisture diffusivity, spherical coordinate movement of moisture, constant temperature and diffusion coefficients and negligible shrinkage during the process of drying is given as the following equation (Doymaz, 2012).

$$MR = \frac{M_t}{M_0} = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp \left[ -\frac{n^2 \pi^2 D_{eff} t}{R^2} \right] \quad (4)$$

$D_{eff}$  is the effective moisture diffusivity ( $m^2 s^{-1}$ ),  $t$  is the time (h),  $R$  denotes the radius of the aril, assumed spherical and constant during the drying period, and  $n$  is a positive integer. With the more extended period of drying, the above equation can be simplified to the only first term of series, without much affecting the accuracy of the prediction (Lopez et al., 2000; Movagharnejad and Nikzad, 2007; Adetoro et al., 2020):

$$\ln(MR) = \ln \frac{6}{\pi^2} - \left( -\frac{\pi^2 D_{eff} t}{R^2} \right) \quad (5)$$

From Eq. (5), a plot of  $\ln MR$  versus drying time give a straight line with a slope ( $K$ ) of

$$K = \frac{\pi^2 D_{eff}}{R^2} \quad (6)$$

### 2.2. Enzyme extraction

Polyphenol oxidase (PPO) and peroxidase (POD) were carried out using a modified method described by Jiang (1999). Dried pomegranate aril (1 g) was grounded into powder and mixed with 10 mL extraction solution (0.1 M phosphate buffer at pH 7, 0.05 M  $L^{-1}$  EDTA and 60 g  $L^{-1}$  polyvinyl polypyrrolidone). The mixture was continuously stirred for 5 min and kept at 4°C for 2 h. Then the mixture was centrifuged at 10,000 rpm for 25 min at 4°C and the supernatant collected and filtered through 0.45  $\mu m$  filter membrane was used as crude enzyme extract.

### 2.2.1. Polyphenol oxidase (PPO)

PPO activity was carried out according to Meighani et al. (2014) with minor modifications. The crude enzymes extract (0.2 mL) was added to 300  $\mu$ L (0.1 M) catechol and 2.5 mL of 0.1 M potassium phosphate buffer (pH 6.0). The absorbance at 420 nm was recorded continuously at 25 °C for 6 min using UV–visible spectrophotometer (Thermo Scientific Technologies, Madison, Wisconsin). The blank sample contained the extraction solution without the enzyme extract. One unit of enzyme activity (U/min/mL) was defined as the amount of enzyme which caused a change of 0.001 in absorbance unit per minute under the conditions of the PPO assay.

### 2.2.2. Peroxidase activity (POD)

POD activity was assayed by a spectrophotometric method using UV–visible spectrophotometer (Thermo Scientific Technologies, Madison, Wisconsin) at 470 nm absorbance. The phenolic substrates used were Guaiacol and hydrogen peroxide as described by Shah et al. (2017) with minor modification. The reaction mixture contained 0.1 mL of 0.045 M guaiacol, 0.15 mL of 0.225 M H<sub>2</sub>O<sub>2</sub>, 2.73 mL of 0.1 M phosphate buffer pH 6 and 20  $\mu$ L of the crude enzyme extract. The blank sample contained the same mixture solution without the enzyme extract. The amount of enzyme which caused a change of 0.001 in absorbance unit per minute under the conditions of the POD assay was referred to as one unit of enzyme activity (U/min/mL). The rate of actual and residual activities was measured in a given time interval. According to Sarpong et al. (2018), the residual enzyme activity was expressed as;

$$REA (\%) = \frac{\text{Current enzyme activity}}{\text{Initial enzyme activity}} \times 100 \quad (7)$$

The residual enzyme activity (REA) gives information about the total reduction of enzymes caused by a heat process. Enzymes inactivation of PPO and POD were investigated by exposing these enzymes to two blanching treatments and further subjected to the same condition of drying. The outcome of the REA described for POD and PPO was given in percentage relative activity.

### 2.3. Colour measurement

Aril colour was determined by direct reading using a chromo-meter (Minolta model CR-200, Osaka, Japan) to obtain the colour values:  $L^*$  (brightness/darkness),  $a^*$  (redness/greenness), and  $b^*$  (yellowness/blueness). The measurements were taken at three different times from a colourless petri dish and averaged. The colour parameters chroma  $C^*$ ,

hue angle  $h^\circ$  and total colour difference ( $\Delta E$ ) were calculated according to Pathare et al. (2013) and Fawole et al. (2013).

$$C^* = (a^{*2} + b^{*2})^{1/2} \quad (8)$$

$$h^\circ = \tan^{-1} (b^*/a^*) \quad (9)$$

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (10)$$

where  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$  are the differences between the colour of the fresh and blanch-assisted dried arils at each treatment levels and were replicated three times. Results were expressed as means  $\pm$  S.E.

## 2.4. Textural profile analysis

The texture of dried pomegranate arils was evaluated using a texture analyzer (TA. XT-Plus, Stable Microsystems Ltd., Surrey, UK), with a 35 mm diameter cylindrical compression probe (Chen and Opara, 2013a,b). Compression test was performed on individual arils with the following operating conditions: pre-test speed 1.5 mm/s, probe test speed 1 mm/s, post-test speed 10.0 mm/s, compression force 10 N. and compression distance 10 mm (Fawole and Opara, 2013). The data obtained from the texture analyzer was interpreted using software Exponent v.4 (Stable Micro System Ltd., Surrey, UK). The software was used to run macro which gave the Hardness (H); positive force (N) measured during compression and Stickiness ( $S_k$ ); negative force (-N) taken before return to the initial position. Dried aril compression test was carried out on 10 randomly selected aril for each drying method, and the results were presented as mean  $\pm$  S.E.

## 2.5. Extraction of samples

Dried pomegranate arils were ground into powder using liquid nitrogen followed by extraction of 5 g sample in 50 mL distilled water. The mixture was vortexed for 5 min and sonicated for 15 min in an ultrasonic bath. This was followed by centrifugation at 10 000 rpm for 25 min, and recovery of the supernatant was used for TSS, TA and pH measurements. For phenolic content and antioxidant capacity, the same procedure was followed using 50 % methanol.

## 2.6. Determination of total soluble solids (TSS), titratable acidity (TA) and pH

TSS was measured using a digital hand refractometer (model PT-32; ATAGO, Tokyo, Japan) with the range of 0-32 °Brix which was blanked with distilled water. For TA, two

millilitres of supernatant was diluted in 70 mL of distilled water and titrated against 0.2 N of sodium hydroxide (NaOH) to a pH of 8.2 using a Metrohm 862 Compact titrosampler (Herisau, Switzerland). BrimA index, a variant of TSS/TA and a criterion for acceptance of fruit juice, which is expressed as  $\text{BrimA} = \text{TSS} - k * \text{TA}$ , where  $k$  is the tongue's sensitivity index normally ranging from 2 - 10 (Fawole and Opara 2013b). In this study  $k$  value of 2 was used to avoid negative BrimA index. The pH value of dried pomegranate aril was measured using a pH meter (Crison, Barcelona, Spain).

## 2.7. Phytochemical properties and antioxidant activity

### 2.7.1. Determination of total phenolic content (TPC)

TPC of dried pomegranate arils was determined by the Folin–Ciocalteu method using a methanolic extract of dried arils. The supernatant (0.05 mL) was mixed with 0.45 mL of 50 % methanol in a test tube followed by the addition of 0.5 mL Folin–Ciocalteu after 2 min. The mixture was then vortexed and kept in the dark for 10 min before adding 2 %  $\text{Na}_2\text{CO}_3$  and further incubation for 40 min in the dark. The absorbance of each sample was read at 520 nm in a UV-visible spectrophotometer (Thermo Scientific technologies, Madison, USA) against a blank containing 50 % methanol. Absorbance was compared with a standard curve (Gallic acid, 0 - 10 mg), and results were expressed as mg gallic acid equivalent per gram pomegranate dry matter (mg GAE/g DM).

### 2.7.2. Total anthocyanin content

Total anthocyanin content (TAC) was quantified using the pH differential method (Wrolstad 1993). In triplicates, extract (1 mL) was mixed with 9 mL of pH 1.0 and pH 4.5 buffers separately. Absorbance was measured at 520 and 700 nm in pH 1.0 and 4.5 buffers, and the result was expressed as cyanidin 3-glucoside using equation 11.

$$A = (A_{510} - A_{700})_{\text{pH } 1.0} - (A_{510} - A_{700})_{\text{pH } 4.5} \quad (11)$$

$$\text{Total monomeric anthocyanin (mg/mL)} = \frac{A \times \text{MW} \times \text{DF}}{\text{xL}}$$

where  $A$ =Absorbance,  $\epsilon$ =Cyd-3-glucoside molar absorbance (26,900),  $\text{MW}$ =anthocyanin molecular weight (449.2),  $\text{DF}$ =dilution factor, and  $L$ =cell path length (1 cm). Final results are expressed as equivalent per gram dry matter (mg C3gE/g DM).

### 2.7.3. Radical-scavenging activity (RSA)

The DPPH assay was carried out in triplicate, according to Fawole et al. (2013). Briefly, under dim light, aqueous methanolic extract of dried aril (0.015 mL) was diluted with methanol



(0.735 mL) in test tubes followed by the addition of methanolic DPPH solution (0.75 mL, 0.1 mM). The mixtures were incubated at room temperature for 30 min in the dark, and the absorbance was measured at 517 nm using a UV–vis spectrophotometer. Absorbance was compared with the standard curve (Trolox equivalent, 0 – 2.0 mM). The free-radical activity of dried aril was expressed as Trolox equivalent (mM) equivalent per gram dry matter (mM TE/g DM).

#### **2.7.4. Ferric ion reducing antioxidant power (FRAP)**

The antioxidant power of dried aril was measured calorimetrically according to Benzie and Strain (1996) and Fawole et al. (2013). The FRAP working solution was freshly prepared in mixtures of 300 mM acetate buffer (50 mL), 10 mM 2,4,6-tripyridyl-s-triazine (TPTZ) (5 mL) and 20 mM ferric chloride (5 mL) at 37 °C. In triplicates, diluted aqueous methanolic dried aril extracts (0.15 mL) were added to 2.85 mL of the FRAP working solution before incubation in the dark for 30 min. The reduction of the  $\text{Fe}^{3+}$ -TPTZ complex to a coloured  $\text{Fe}^{2+}$ -TPTZ complex at low pH by dried aril extracts was monitored by measuring the absorbance at 593 nm. Trolox (0 - 10 mM) was used for the calibration curve, and the results were expressed as Trolox (mM) equivalents per gram dry matter (mM TE/g DM).

### **2.8. Statistical analysis**

The analyses made from the chemical properties, colour and phytochemical properties were subjected to statistical evaluation. Data were processed with STATISTICA (Statistica 13.0, StatSoft Inc., Tulsa, OK, USA) and presented as mean  $\pm$  standard error. All analysis was done in triplicates. Data was subjected to analysis of variance (ANOVA), and means were separated according to Fisher's LSD test at a level of significance of 95% confidence level. GraphPad Prism software 4.03 (GraphPad Software, Inc., San Diego, USA) was used for graphical presentations. Principal component analysis (PCA) was carried out using the XLSTAT software version 2012.04.1 (Addinsoft, France).

## **3. Results and Discussion**

### **3.1. Effect of blanching treatments on drying kinetics of dried pomegranate arils**

The characteristics drying curves showing changes in the moisture ratio of blanched and unblanched dried arils with drying time at temperature 60°C, 19.6 % RH and 1.0 m s<sup>-1</sup> constant air velocity is illustrated in (Fig. 1a). Moisture ratio decreased continuously with drying time as expected. According to the result, moisture decreased rapidly in blanched arils, and the desired moisture was reached faster in both blanched arils (420 min), while unblanched

arils (660 min). Hence, blanched arils reduced drying time by approximately 36.4 %. This was in agreement with Thakur et al. (2010) for blanched pomegranate arils and Doymaz (2010) for blanched Amasya red apples. This suggested a positive impact of blanching by reducing the drying time in pomegranate arils. Similar results were also reported by other researchers, for instance, Karaaslan et al. (2014) pre-treated pomegranate arils under vacuum dryer, while Sarpong et al. (2018) reported approximately 20% reduction in the time of drying for pre-treated dried banana.

Figure 1b showed the changes in the drying rate as a function of drying time for the same drying treatments. Drying rates for both blanched and unblanched arils followed the usual trend of falling rate period. This phenomenon could be considered as a diffusion-controlled process where the rate of eliminating moisture is by diffusion, the permeability of moisture from inside of the material to its surface. A similar trend was observed in the studies by Kaya et al. (2007) in apple pomace and Kingsly et al. (2007) in dried peach fruit. Furthermore, both blanched treatments commenced their shrinkage at higher drying rates than the unblanched arils. For instance, the highest drying rate was observed for arils treated at 90°C for 30s followed by 100°C for 60s (0.363 and 0.353 g min<sup>-1</sup>), respectively, (Fig. 1b) while unblanched (control) arils had the least values (0.311 g min<sup>-1</sup>) of drying rate. This is an indication that blanching treatments increases the drying rate for pomegranate arils. The high rate of drying is related to the differences in microstructure of arils due to the level of membrane shrinkage and high pore spaces during blanching. Also, pretreatments, either mechanical, chemical or heat could be targeted in part, to reduce the drying time of the product by exposing the pore spaces to increase the movement of moisture from the internal region (Tunde-Akintunde and Ogunlakin, 2011). The result from this study is in accordance with the study by Wang et al. (2007) who reported a microstructural difference between fresh apple pomace and the pre-treated apple pomace due to the level of porosity and shrinkage.

The values of moisture effective diffusion ( $D_{\text{eff}}$ ) were shown in Table 1. For blanched arils, the  $D_{\text{eff}}$  values were  $1.09 \times 10^{-8}$  and  $1.29 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$  at 90°C for 30s and 100°C for 60s respectively, while the unblanched arils were  $5.83 \times 10^{-9} \text{ m}^2 \text{ s}^{-1}$ . In general, higher values of  $D_{\text{eff}}$  were observed in the blanched arils and higher than those obtained for unblanched arils, which indicates faster removal of moisture and faster drying of the arils. Zogzas et al. (1996) reported that values of moisture diffusivity in food drying experiments are within the range of  $10 \times 10^{-12}$  to  $10 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$ , our values for moisture diffusivity are within the range observed in this study. However, the values of moisture diffusivity in our study were farther from the

range previously reported for vacuum dried pomegranate arils ( $0.74 \times 10^{-10}$  to  $5.25 \times 10^{-10} \text{ m}^2 \text{ s}^{-1}$ ) and microwave dried arils ( $3.43 \times 10^{-10}$  to  $3.05 \times 10^{-10} \text{ m}^2 \text{ s}^{-1}$ ) (Minaei et al., 2012). Also,  $D_{\text{eff}}$  values reported by Doymaz (2012) for dried pomegranate arils ranged between  $9.373 \times 10^{-11}$  to  $3.429 \times 10^{-10} \text{ m}^2 \text{ s}^{-1}$ .

### 3.2. Influence of blanching on enzymes inactivation

The result of blanching on the polyphenol oxidase and peroxidase are presented in the (Fig. 2). A significant ( $p < 0.05$ ) difference was observed among the blanching treatments for the residual PPO activity (Fig. 2a). Blanching reduced the effect of enzyme activity by 76 and 68 % for both blanching treatment at  $90^\circ\text{C}$  for 30s and  $100^\circ\text{C}$  for 60s, respectively, compared to unblanched arils with the highest value of residual enzyme activity. Similarly, POD inactivation was significantly ( $p < 0.05$ ) different among blanching treatments (Fig. 2b). Unblanched arils had the highest residual POD activity (14.9 %), followed by  $100^\circ\text{C}$  60s (3.52 %), while arils blanched at  $90^\circ\text{C}$  30s had the least value (1.25 %). The trend observed for both residual enzyme activity (PPO and POD) was more effective for blanched arils treated at  $90^\circ\text{C}$  for 30s and  $100^\circ\text{C}$  60s. This could be related to a higher blanching temperature (90 and  $100^\circ\text{C}$ ) and exposure time (30 and 60s) which is known to reduce enzyme activity in pomegranate arils. The synergistic effect of pretreatment and drying was observed to reduce enzyme activities in banana slices due to higher temperature (Sarpong et al., 2018). A similar result was observed in this study, where both blanching treatments ( $90^\circ\text{C}$  for 30s and  $100^\circ\text{C}$  for 60s) reduced enzyme activity. The mechanism to the result was reported by Jaiswal et al. (2010). The authors noted that in pre-treated food materials, quinones are formed from phenolic compounds due to enzyme inactivation as a result of the presence of hydrogen peroxide appearing in a low peroxidase level.

### 3.3. Colour change

Blanching retained the colour attributes of dried pomegranate arils significantly ( $p < 0.05$ ). From Table 2, blanched arils at both  $90^\circ\text{C}$  for 30s (26.5) and  $100^\circ\text{C}$  for 60s (24.6) had better retention of lightness ( $L^*$ ) with closer values to the fresh arils (29.4) compared to unblanched dried arils (21.4). The blanched arils at  $90^\circ\text{C}$  for 30s and  $100^\circ\text{C}$  for 60s were 9.8 and 16.3 % darker, respectively than the fresh arils, while unblanched arils (27 %) darker than the fresh arils. For pomegranate dried aril redness ( $a^*$ ), blanched arils at  $90^\circ\text{C}$  for 30s and  $100^\circ\text{C}$  for 60s as well as fresh arils had significantly lower values (17.6, 18.4 and 18.4), respectively, compared to unblanched arils (20.4). This also suggests that blanched arils maintained the red colour of dried arils compared to fresh arils. This may be an indication that

blanching helps retain anthocyanin pigments, thereby reducing browning (Deylami et al., 2016). Also, the reduction in enzymatic activity (PPO and POD) in blanched arils (Fig. 2) could be related to the retention of red colour attribute in dried pomegranate arils. The significant increase in  $a^*$  value for the unblanched arils was expected. This could in part, be due to pigment instability with regards to high sensitivity of fruit to heat application or enzymatic browning reaction or both during dehydration (Ashebir et al., 2009; Vega-Galvez et al., 2008).

The chroma ( $C^*$ ) of dried pomegranate arils showed the degree of colour intensity. Blanched arils at 90°C for 30s and 100°C for 60s had the lowest values for colour intensity (18.2 and 18.9), respectively, compared to the unblanched arils (22.0) (Table 2). This implies that  $C^*$  contributed to the degree of redness of dried arils for both blanched arils 90°C for 30s and 100°C for 60s. Further, the process of enzyme inactivation as a result of blanching retained the colour intensity of dried arils. Our results agreed with Deylami et al. (2016), who reported high retention in  $a^*$  and  $C^*$  of blanched mangosteen due to PPO inactivation. Similarly, hue angle showed the highest values for fresh arils followed by the unblanched arils, while 90°C for 30s and 100°C for 60s had the lowest hue values. Colour retention is known as one of the quality indicators to evaluate the extent of deterioration due to thermal processing (Horuz and Maskan, 2015). The total colour difference (TCD) was significantly higher for unblanched arils (8.56) compared to 90°C 30s (6.77) and 100°C 60s (7.14) (Table 2). This implies that blanched arils at 90°C for 30s and 100°C for 60s in this study had a better appearance considering the lower TCD values than the unblanched arils. Colour is an important factor affecting marketability and consumer preference of fruit (Opara et al., 2009; Pathare et al., 2012), blanched arils would be more preferred than the unblanched arils.

### **3.4. Total soluble solids (TSS), Titratable acidity (TA) and pH**

Chemical properties such as TSS, TA and TSS/TA have been used to describe taste (flavour) with regards to the sweetness and acidity; these properties have also been used as a quality criterion for the formulation of pomegranate products (Al-Said et al., 2009). As shown in Table 3, all chemical properties measured for dried pomegranate arils significantly ( $p < 0.05$ ) increased by blanching treatment. TSS increased from 16.1 °Brix to 24.9 °Brix for unblanched arils after drying, followed by blanched arils at 90°C for 30s (21.4 °Brix). The least TSS amongst the treated arils was recorded for arils blanched at 100°C for 60s (18.5 °Brix). In other words, a lower temperature (90 °C) and time exposure time (30 s), resulted in higher total soluble content compared to arils treated at higher temperature and time of 100°C for 60s. This could be as a result of leaching during blanching process which could be more at a higher

temperature (100°C) and longer time of exposure (60s). However, the significant increase in TSS for the unblanched arils could be attributed to the fact that there was no pretreatment at any stage before drying; hence, soluble solids within the aril sac were tightly packed together.

Titrateable acidity (TA, expressed as % citric acid) was significantly ( $p < 0.05$ ) increased in the blanched and unblanched arils (Table 3). Unblanched arils had the highest values for TA, followed by arils at 90°C for 30s. Fresh arils had the least TA values. A significant ( $p < 0.05$ ) change in citric acid with approximately 65 % increase in citric acid was observed for unblanched arils compared to fresh arils, while arils blanched at 90°C for 30s and 100°C for 60s increased (58 and 56 %), respectively, as compared with fresh arils. However, amongst the treatments, blanched arils had the least values of TA compared to unblanched aril. Several studies have reported that organic acids are produced within the fruit from stored carbohydrate materials (Sakiyama and Stevens 1976; Workneh et al. 2014). The decrease in TA in the blanched arils could be due to the conversion of acid to carbohydrate through an extended time of heat treatment. Our results are in agreement with those reported by Brennard (1994), who observed a decrease in TA due to extended drying time in sun-dried fruit. As a result of changes in TSS and TA contents, the values of TSS/TA decreased considerably for both blanched and unblanched arils compared to fresh arils. However, there was no statistical difference amongst the treatments. Unblanched arils had 5.95 TSS/TA ratio, while arils blanched at 90°C for 30s and 100°C for 60s had TSS/TA ratio of 6.01 and 5.45, respectively. The TSS/TA ratio of blanched pumpkin fruit slices after drying ranged between 3.4 and 4.3 (Workneh et al. 2014), which was lower than the values generated for dried pomegranate arils. TSS/TA value is an important criterion also for evaluating the quality of pomegranate fruit products (Al-Said et al., 2009). Results obtained in this study could be appropriated to the highest proportion of sugar to acid ratio after thermal treatments.

To further explore the relationship between TSS and TA as a potential chemical indicator related to flavour, BrimA index was calculated according to Jordan et al. (2001) (Table 3). BrimA significantly ( $p < 0.05$ ) increased in unblanched (16.6) and blanched (14.3) arils that were treated at 90°C for 30s, compared to the blanched arils treated at 100°C for 60s (11.7). The increase in BrimA index for pomegranate dried arils could be as a result of the small amount of acid to sugar ratio observed in this study. Jaya and Das (2003) reported that in the calculation of BrimA index, the same numerical changes are observed as a result of the minimal amount of acid than sugar. The pH of fruit usually characterizes its acidic taste (Fawole and Opara 2013b). A significant ( $p < 0.05$ ) increase in dried aril pH after drying was

observed amongst the treatments (Table 3). Higher pH was observed in blanched arils at 90°C for 30s and 100°C for 60s compared to the unblanched arils. The increase in the values of pH for blanched pomegranate arils could be due to the change in an organic acid to carbohydrate because of the extensive thermal treatment. Workneh et al. (2014) reported that changes observed in the pH of food materials during the period of heat treatment were due to the synthesis of organic acids from carbohydrates.

### **3.5. Textural properties**

Dried aril hardness increased with or without blanching as presented in Table 4, indicating that the amount of force used to compress unblanched dried arils was similar to that of blanched arils. Arils blanched at 90°C for 30s, and 100°C for 60s had lower values of hardness (5448.9 and 5339.7 N), respectively, than unblanched arils (5633.1 N). Differences in the values of hardness between unblanched and blanched arils could partly, be due to the rate at which moisture evaporated from the arils or increased concentration of solids in the arils after drying contributing to the mechanical strength of the albedo (seed). Overall hardness in dried arils after processing suggests that dried arils were not affected by blanching. Results from our study were in contrast to those reported by Akpinar et al. (2003), who observed a loss of firmness (hardness) in blueberry after the thermal process.

Furthermore, the force required for the stickiness of blanched arils to return to its initial position was significantly ( $p < 0.05$ ) different from the unblanched aril (Table 4). The force required for the stickiness of blanched arils decreased with increase in temperature and extended time. For instance, blanching at 100°C for 60s was less sticky (-23.3 N) than 90°C for 30s (-35.1 N). This observation indicated that temperature (90 and 100 °C) and extended time (30 and 60 s) during blanching results in an increase in dried aril stickiness. However, unblanched arils were stickier (-46.8 N) than the blanched arils. The textural dynamics shown in the sticky product could be as a result of high sugar content observed around the dried arils after thermal treatment.

### **3.6. Total phenolic content (TPC)**

There was a significant ( $p < 0.05$ ) difference in the TPC of dried pomegranate arils (Fig. 3). A decline in TPC was generally observed among the blanching treatments compared to the fresh arils. This could be due to thermal application on the arils. Rawson et al. (2011) noted that the influence of drying or thermal processing reduces the stability of the bioactive component in food materials. However, TPC was higher in blanched arils compared to the



unblanched arils. Both blanched pomegranate arils 90°C for 30s and 100°C for 60s had higher TPC (148.8 and 141.7 mg GAE/g DM), respectively than unblanched arils with the least TPC (102.1 mg GAE/g DM). Differences observed in the values of TPC for blanched and unblanched arils in this study suggested the level of sensitivity of TPC to temperature during the thermal treatment. Fazaeli et al. (2013) noted that bioactive compound such as phenolic and anthocyanin concentration are highly heat-sensitive during thermal processing. Similar results were reported by Karaaslan et al. (2014), in which pretreatment resulted in a significant change in the phenolic content of pomegranate arils.

### 3.7. Total anthocyanin content (TAC)

TAC in pomegranate fruit is responsible for the characteristic red colouration (Gil et al., 1996; Artés et al., 1998; Fawole and Opara, 2013). A general decrease in TAC was noticed among the treatments compared to the fresh arils. TAC in the blanched arils treated at 90°C for 30s retained more TAC in comparison with the unblanched arils. However, TAC in blanched arils treated at 100°C for 60s was not statistically ( $p < 0.05$ ) different from unblanched arils. Dried pomegranate arils blanched at 90°C for 30s had the highest TAC (28.6 mg C3gE/g DM), followed by 100°C for 60s (24.8 mg C3gE/g DM) while unblanched arils (21.8 mg C3gE/g DM) (Fig. 4). TAC of dried pomegranate arils blanched at 90°C for 30s was higher by approximately 23.8 %, respectively, compared to the unblanched arils. Sablani et al. (2011) reported that blanching treatment before air-drying increased the retention of phenolic and total anthocyanin content in dried berries which is in agreement with the findings from our study. Furthermore, the high total anthocyanin content for blanched arils at 90°C for 30s could be attributed to the increase in biosynthesis and accumulation of anthocyanin which is known to be induced in pomegranates at lower temperatures (Miguel et al., 2004). Increase in redness ( $a^*$ ) colour of a dried aril in this study could be related directly with high total anthocyanin content.

### 3.8. Antioxidant capacity

The RSA of dried pomegranate arils was significantly ( $p < 0.05$ ) higher in blanched arils compared to unblanched arils as presented in (Fig. 5a). RSA of dried arils was in the order of 90°C for 30s (32.0 mM TE/g DM) > 100°C for 60s (31.1 mM TE/g DM) and unblanched (17.0 mM TE/g DM) (Fig. 5a). Higher RSA for blanched arils of dried pomegranate arils could be an indication of high retention of antioxidant capacity after thermal treatment. Blanching treatment enhances the retention of the phytochemicals as reported by Karaaslan et al. (2014). Similarly, Sablani et al. (2011) reported higher content of phytochemicals in blanched berries

before air drying. Among the blanching treatments in the FRAP of dried arils, the highest FRAP was found in the blanched arils at 90°C for 30s (4.92 mM TE/g DM), while unblanched arils had the least value (2.14 mM TE/g DM) (Fig. 5b). However, the amount of FRAP in blanched arils treated at 100°C for 60s (4.49 mM TE/g DM) was not statistically ( $p < 0.05$ ) different from unblanched arils. A similar result was reported by Nurhuda et al. (2013) that blanching caused no significant difference in the antioxidant capacity of the final product.

### **3.9. Multivariate analysis**

#### **3.9.1. Principal component analysis**

The results show the average of enzyme activity, phenolics, antioxidant capacity, chemical attributes and colour coordinates of blanched and unblanched pomegranate dried arils. The two principal components (F1 and F2) explains 100.0 % of the total data variance (Fig. 6). As observed, F1 explained 86.49 % of the total variability among blanched and unblanched dried pomegranate arils while F2 explained only 13.51 % as described in Fig. 6. The observations (Fig. 6) indicated that dried arils blanched at 90°C for 30s and 100°C for 60s could be associated with  $L^*$ , pH, TPC, TAC, FRAP and RSA which had higher positive scores along F1 (Table 5). Moreover, the higher negative scores (Table 5) along F1 (Fig. 6) corresponds to hue, TCD,  $a^*$ , chroma  $C^*$ , BrimA, TSS, PPO, POD and TA for the unblanched arils. Also, lower negative scores along F1 were for unblanched arils (associated with TSS/TA). On the other hand, high positive scores (Table 5) along F2 were associated with TSS/TA and TAC for unblanched and blanched arils at 90°C for 30s, respectively, (Fig. 6). Likewise, for high negative value along F2 was blanched arils at 100°C for 60s (pH). Similarly, along the F2, low positive scores (as shown in Fig. 6 and Table 5) for unblanched and blanched arils (90°C for 30s) could characterize for hue, TSS, TA, BrimA as well as  $L^*$ , TPC and FRAP, respectively. However, lower negative scores along F2 were for unblanched arils (associated with hue, TCD, PPO, POD,  $a^*$  and  $C^*$ ) as well as RSA for blanched arils at 100°C for 60s. The results demonstrated on the PCA showed that blanched and unblanched treatments for pomegranate dried arils have significantly different properties.

#### **3.9.2. Correlation between quality attributes of dried arils**

Pearson's correlation was used to investigate the interrelationships between quality attributes of blanched and unblanched arils which includes enzyme activity, chemical and colour attributes as well as phenolic contents and antioxidant capacity (Table 6). Significantly ( $p < 0.05$ ) strong relationships were revealed among some of the quality attributes evaluated. TSS and TA showed strong positive correlation ( $r = 0.966$ ). This relationship clearly showed



that increase in dried aril TA might also bring about an increase in TSS in blanched arils. Lightness ( $L^*$ ) showed a strong negative correlation with enzyme activities (PPO and POD) ( $r = -0.998$  and  $-1.000$ ), respectively. This relationship indicates that the higher the  $L^*$  of blanched arils, the lower the enzyme activity. This suggested that blanching halted the effect of browning in aril colour after drying. Another interesting relationship was the strong negative correlation between  $a^*$  and TAC. This suggests that decrease in the redness of pomegranate arils during drying increases the TAC, as against scientific evidence reported in many studies of pomegranate fruit Fawole et al. (2013) and Arendse et al. (2014) contributing to better red colouration as one of the desired attribute in pomegranate marketing. With regards to the reported health benefits of consuming dried pomegranate arils, high in phenolic compounds (Thakur et al., 2010), it is therefore not surprising that antioxidant capacity (RSA and FRAP) showed a positive correlation with TPC and TAC. Therefore, the result from our correlation matrix showed plausible retention of antioxidant capacity of blanched arils and also highly dependent on the TPC.

#### 4. Conclusion

The effect of blanching treatments (90°C for 30s and 100°C for 60s and unblanched (control)) on the drying kinetics, enzyme inactivation and other qualities of dried pomegranate arils were studied. Both blanching treatments (90°C for 30s and 100°C for 60s) had a higher drying rate and reduced drying time. However, the results obtained showed that blanching treatment at 90°C for 30s reduced the polyphenol oxidase and peroxidase enzyme activity significantly compared to those treated at 100°C for 60s. It was also observed that blanching exhibited differences in the physicochemical and phytochemical properties of dried pomegranate aril. For instance, unblanched arils significantly increased the total colour difference, TSS, TA and BrimA of pomegranate dried arils. However, lightness ( $L^*$ ) of dried arils was higher at 90°C for 30s and corresponded considerably with the total anthocyanin content. Blanching treatment (90°C for 30s) retained the phytochemical properties of dried arils. However, arils blanched at 90°C for 30s had a significantly higher total anthocyanin content in. The evaluated quality components reported in this study provides scientific evidence that could be used towards drying processes, enzymatic inactivation, phytochemical and antioxidant profiling of dried pomegranate arils which could serve as an objective guide in the choice of blanching treatment for dried aril processing. Hence, blanching at a lower temperature (90°C) and shorter time (30s) is suggested to process and optimally retain the biochemical qualities of dried pomegranate arils.

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**Table 1**Moisture effective diffusivity ( $D_{\text{eff}}$ ) of dried pomegranate arils for different blanching conditions

Blanching condition	Moisture diffusivity ( $\text{m}^2/\text{s}$ )
Control	$5.83 \times 10^{-9}$
90°C, 30s	$1.09 \times 10^{-8}$
100°C, 60s	$1.29 \times 10^{-8}$

**Table 2**

Changes in colour attributes of fresh and dried pomegranate arils for different blanching conditions

Blanching condition	L*	a*	C*	h°	TCD
Fresh	29.4±0.06 <sup>a</sup>	18.4±0.25 <sup>b</sup>	21.2±0.22 <sup>a</sup>	29.8±1.51 <sup>a</sup>	-
Control	21.4±0.08 <sup>c</sup>	20.4±0.08 <sup>a</sup>	22.0±0.02 <sup>a</sup>	21.9±0.63 <sup>b</sup>	8.56±0.16 <sup>a</sup>
90°C, 30s	26.5±0.67 <sup>b</sup>	17.6±0.81 <sup>b</sup>	18.2±0.85 <sup>b</sup>	14.7±1.37 <sup>c</sup>	6.77±0.68 <sup>b</sup>
100°C, 60s	24.6±0.56 <sup>b</sup>	18.4±0.19 <sup>b</sup>	18.9±0.14 <sup>b</sup>	13.6±1.83 <sup>c</sup>	7.14±0.10 <sup>b</sup>

L\*, lightness; a\*, redness; C\*, chroma; h°, hue angle; TCD, total colour difference. Data presented as means ± SE in each row followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.

**Table 3**

Chemical attributes of fresh and dried pomegranate arils with different blanching conditions

Blanching condition	TSS (°Brix)	TA (% citric acid)	TSS/TA	pH	BrimA
Fresh	16.1±0.18 <sup>d</sup>	1.47±0.06 <sup>c</sup>	11.0±0.39 <sup>a</sup>	3.22±0.16 <sup>c</sup>	13.2±0.13 <sup>c</sup>
Control	24.9±0.37 <sup>a</sup>	4.20±0.06 <sup>a</sup>	5.95±0.16 <sup>b</sup>	3.39±0.01 <sup>bc</sup>	16.6±0.47 <sup>a</sup>
90°C, 30s	21.4±0.10 <sup>b</sup>	3.57±0.09 <sup>b</sup>	6.01±0.17 <sup>b</sup>	3.56±0.01 <sup>ab</sup>	14.3±0.26 <sup>b</sup>
100°C, 60s	18.5±0.15 <sup>c</sup>	3.40±0.06 <sup>b</sup>	5.45±0.09 <sup>b</sup>	3.75±0.01 <sup>a</sup>	11.7±0.15 <sup>d</sup>

TSS, total soluble solids; TA, titratable acidity. Data presented as means ± SE in each row followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.



**Table 4**

Changes in the hardness and stickiness of dried pomegranate arils

Blanching condition	Hardness (N)	Stickiness (-N)
Fresh	117.1±1.09 <sup>b</sup>	-
Control	5633.1±233.8 <sup>a</sup>	-46.8±3.68 <sup>c</sup>
90°C 30s	5448.9±221.1 <sup>a</sup>	-35.1±4.21 <sup>b</sup>
100°C 60s	5339.7±209.1 <sup>a</sup>	-23.3±2.69 <sup>a</sup>

Data presented as means ± SE in each row followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.

**Table 5**

Factor loadings and score for the first two principal (F1–F2) components of dried pomegranate arils at different blanching conditions.

Loadings	F1	F2
L*	0.981	0.196
a*	-0.950	-0.312
C*	-0.976	-0.218
h°	-0.995	0.098
TCD	-0.975	-0.224
pH	0.865	-0.501
TSS	-0.909	0.417
TA	-0.986	0.167
TSS/TA	-0.440	0.898
BrimA	-0.867	0.498
PPO	-0.992	-0.130
POD	-0.983	-0.185
TPC	0.998	0.057
TAC	0.814	0.581
RSA	1.000	-0.001
FRAP	0.989	0.145
Scores		
90°C 30s	2.490	1.832
100°C 60s	2.769	-1.768
Control	-5.258	-0.064

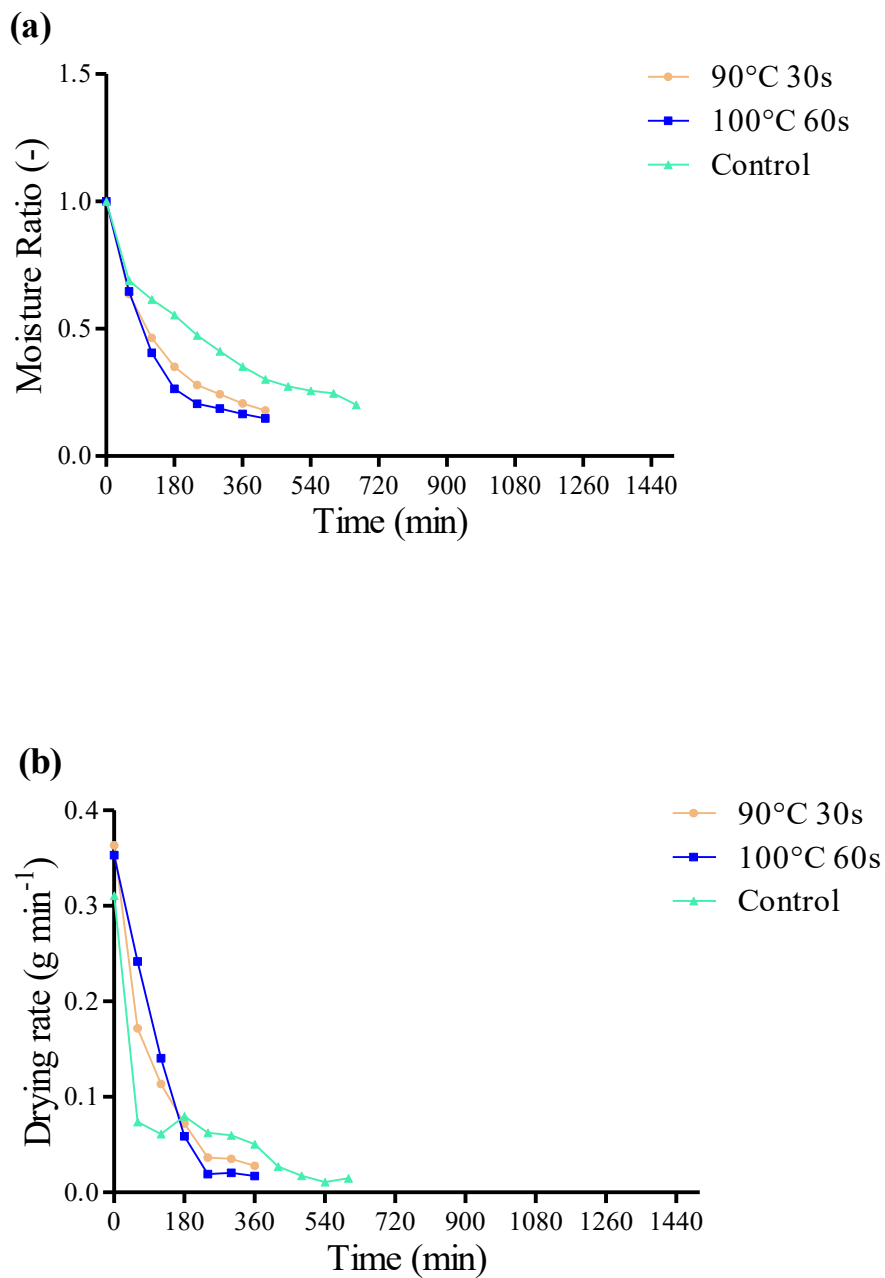
L\*, lightness; a\*, redness; C\*, chroma; h°, hue angle; TCD, total colour difference; RSA, radical scavenging activity; FRAP, ferric reducing antioxidant power; TPC, total phenolic content; TAC, total anthocyanin content; PPO, polyphenol oxidase; POD, peroxidase; TSS, total soluble solids; TA, titratable acidity.

**Table 6**

Pearson's correlation coefficients (r) among the investigated parameters at different blanching conditions

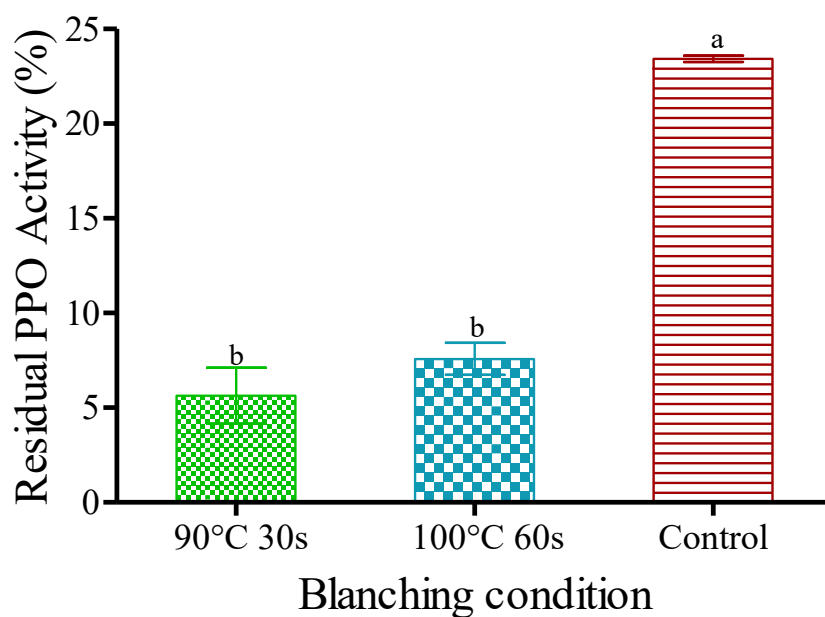
Variables	L*	a*	C*	h°	TCD	pH	TSS	TA	TSS/TA	Brim A	PPO	POD	TPC	TAC	RSA	FRAP
L*	<b>1</b>															
a*	-0,993	<b>1</b>														
C*	<b>-1,000</b>	0,995	<b>1</b>													
h°	-0,957	0,915	0,950	<b>1</b>												
TCD	<b>-1,000</b>	0,996	<b>1,000</b>	0,948	<b>1</b>											
pH	0,750	-0,666	-0,735	-0,910	-0,731	<b>1</b>										
TSS	-0,809	0,734	0,796	0,945	0,792	-0,996	<b>1</b>									
TA	-0,934	0,885	0,926	<b>0,998</b>	0,923	-0,937	0,966	<b>1</b>								
TSS/TA	-0,255	0,139	0,234	0,526	0,228	-0,831	0,775	0,584	<b>1</b>							
Brim A	-0,753	0,669	0,738	0,912	0,734	<b>-1,000</b>	0,996	0,938	0,829	<b>1</b>						
PPO	<b>-0,998</b>	0,983	0,996	0,974	0,995	-0,793	0,847	0,956	0,320	0,795	<b>1</b>					
POD	<b>-1,000</b>	0,991	<b>0,999</b>	0,960	<b>0,999</b>	-0,758	0,816	0,938	0,267	0,761	<b>0,998</b>	<b>1</b>				
TPC	0,990	-0,966	-0,987	-0,988	-0,986	0,835	-0,884	-0,975	-0,388	-0,838	<b>-0,997</b>	-0,992	<b>1</b>			
TAC	0,912	-0,955	-0,921	-0,754	-0,924	0,413	-0,498	-0,705	0,163	-0,417	-0,883	-0,907	0,846	<b>1</b>		
RSA	0,980	-0,950	-0,976	-0,995	-0,974	0,866	-0,909	-0,986	-0,441	-0,868	-0,991	-0,983	<b>0,998</b>	0,814	<b>1</b>	
FRAP	<b>0,999</b>	-0,985	<b>-0,997</b>	-0,971	-0,997	0,784	-0,839	-0,951	-0,306	-0,786	<b>-1,000</b>	<b>-0,999</b>	0,996	0,890	0,989	<b>1</b>

Correlation values in **bold** are significant at  $p < 0.05$ . TPC, total phenolic content; TAC, total anthocyanin content; RSA, radical scavenging activity; FRAP, ferric reducing antioxidant power; PPO, polyphenol oxidase; POD, peroxidase; L\*, lightness; a\*, redness; C\*, chroma; h°, hue angle; TCD, total colour difference; TSS, total soluble solids; TA, titratable acidity.

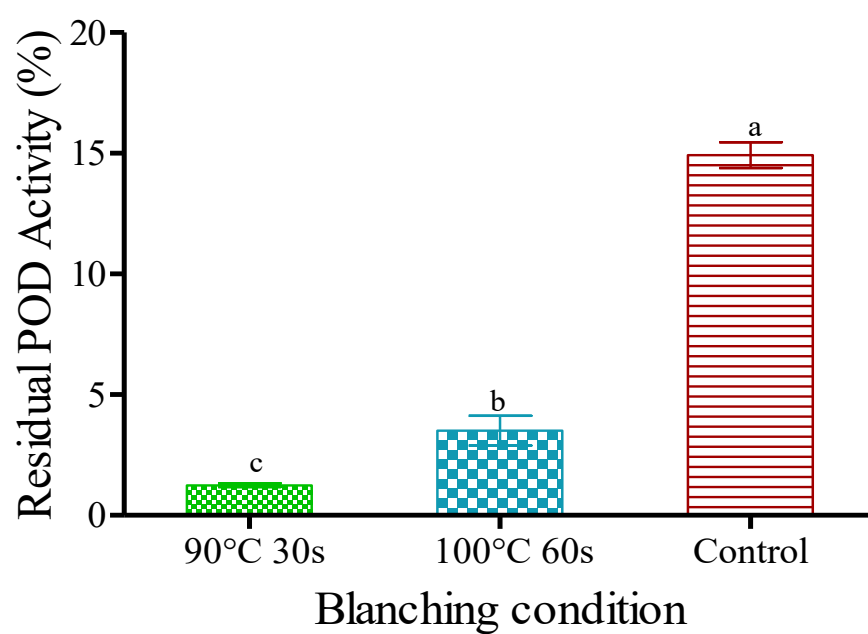


**Fig. 1.** Variation in (a) moisture ratio and (b) drying rate for different blanching conditions versus drying time

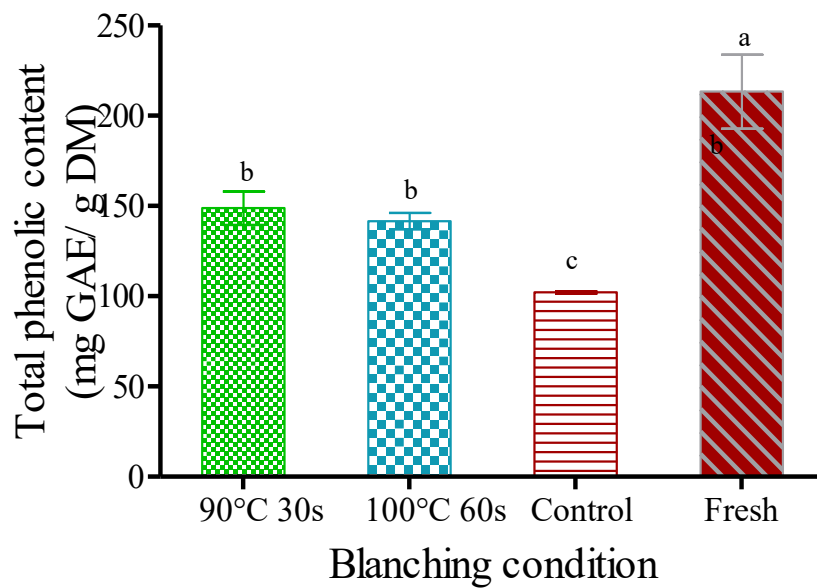
(a)



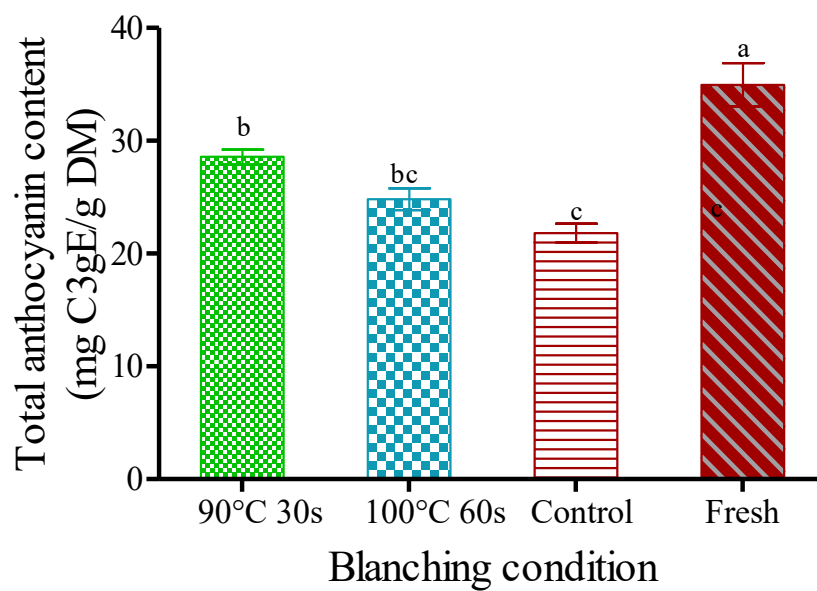
(b)



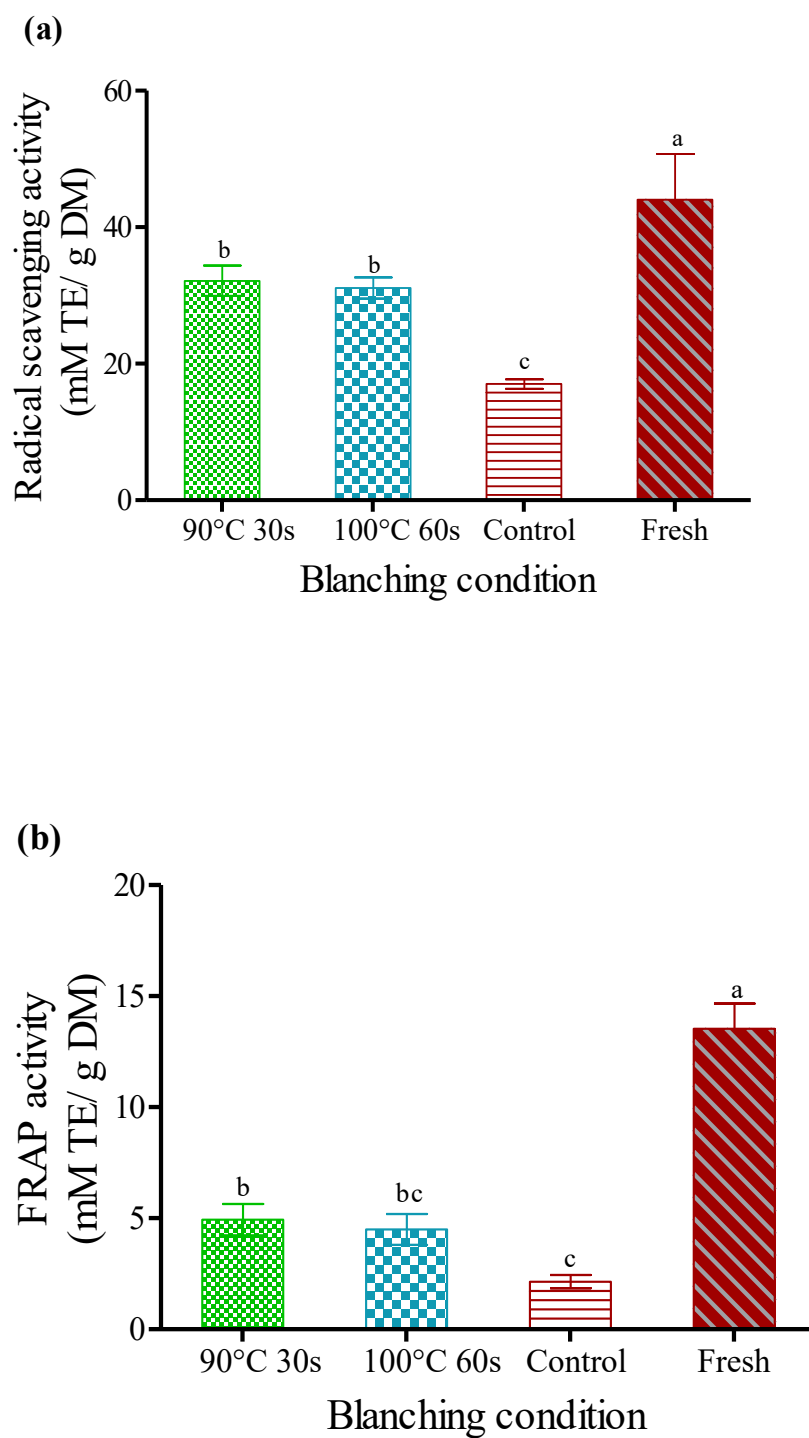
**Fig. 2.** Effects of blanching conditions on the residual (a) polyphenol oxidase (PPO) and (b) peroxidase (POD) of dried pomegranate aril cv. Wonderful



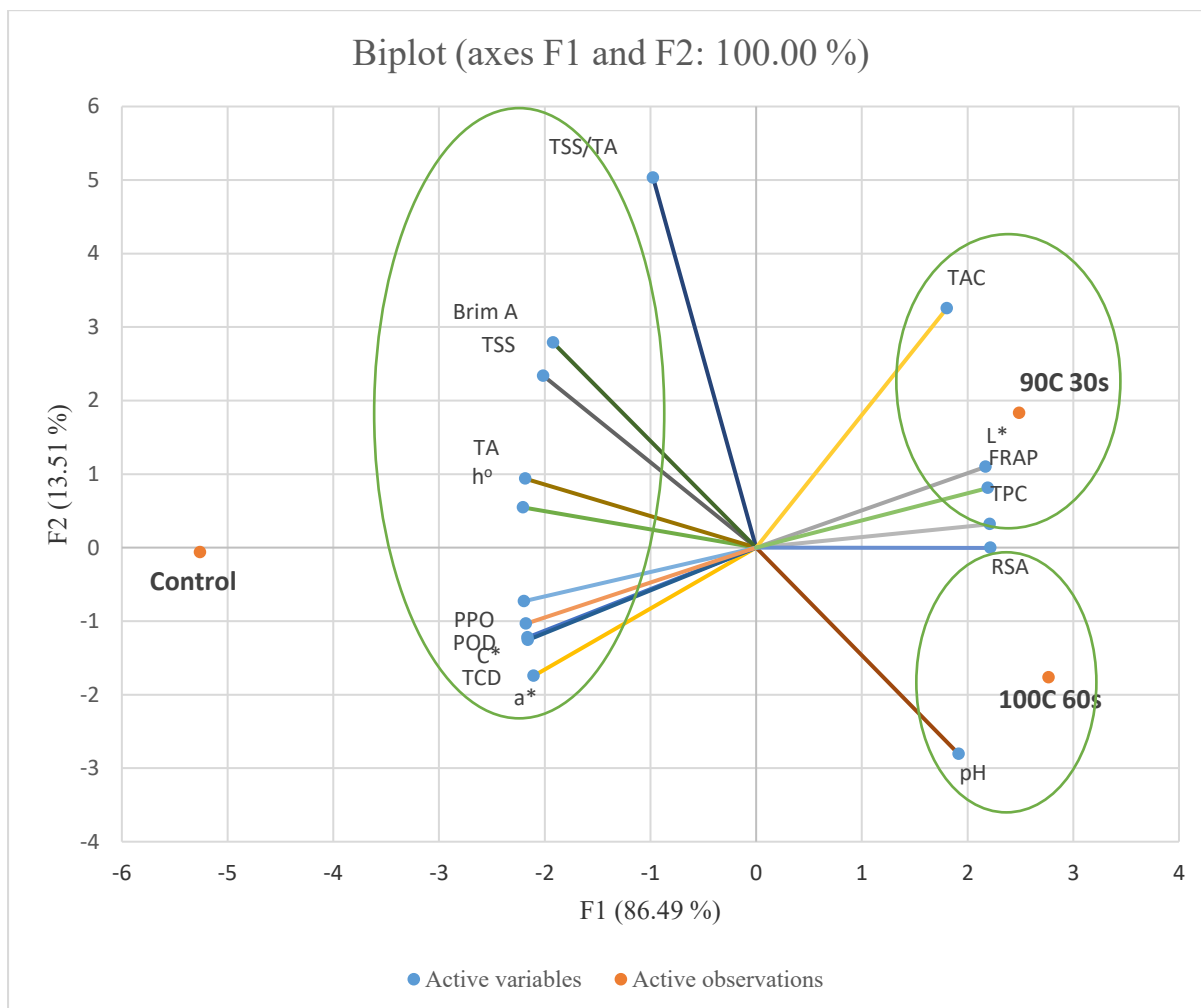
**Fig. 3.** Total phenolic content of dried pomegranate arils cv. Wonderful at different blanching conditions (mg GAE/ g DM). Fresh arils in mg/100 mL.



**Fig. 4.** Total anthocyanin content in dried pomegranate arils cv. Wonderful at different blanching conditions. Fresh arils in mg/100 mL.



**Fig. 5.** Antioxidant capacity (a) radical-scavenging activity (RSA) and (b) Ferric ion reducing antioxidant power (FRAP) activity of dried pomegranate arils cv. Wonderful at different blanching conditions. Fresh arils in mg/100 mL.



**Fig. 6.** Principal component analysis of the first two factors (F1 and F2) based on physicochemical properties, phenolic content and antioxidant activities of dried pomegranate arils cv. Wonderful at different blanching conditions. L\*; lightness/darkness, a\*; redness/greenness, C\*; chroma, h°; hue angle, TCD, total colour difference, RSA, radical scavenging activity; FRAP, ferric reducing antioxidant power, TPC, total phenolic content; TAC, total anthocyanin content; PPO, polyphenol oxidase; POD, peroxidase; TSS, total soluble solids; TA, titratable acidity.



## THEME D

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### **Pomegranate juice powder development and storability**

- Effects of carrier agents on the biochemical activities and rheological properties of freeze-dried pomegranate juice (*Punica granatum*) powder (Paper 6)
  - Influence of packaging materials on the storage stability of biochemical composition of freeze-dried pomegranate (*Punica granatum*) juice powder (Paper 7)
-

## PAPER 6

### Effect of carrier agents on the physicochemical, antioxidant capacity and rheological properties of freeze-dried pomegranate juice (*Punica granatum*) powder

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#### ABSTRACT

The physicochemical, antioxidant capacity and rheological properties of freeze-dried ‘Wonderful’ pomegranate juice powder (PJP), produced with different carrier agents, was investigated. Powders were produced using maltodextrin, gum arabic and waxy starch as carrier agents and characterized by scanning electron microscopy and particle size distribution. Results showed that PJP produced with maltodextrin had the highest yield (46.6%), followed by gum arabic (40.6%), while, waxy starch had the least yield (35.4%). Powders produced with maltodextrin (96.5%) and gum arabic (96.1%) were highly soluble, which indicates better reconstitution properties. Waxy starch-added PJP had the least hygroscopicity (4.7%) which offers good stability during storage and lower degree of caking, compared to maltodextrin (10.2%) and gum arabic (12.6%) powders. Freeze-dried pomegranate powder produced with maltodextrin retained more redness ( $a^*$ ) by approximately 44 %, compared to gum arabic. Similarly, PJP with maltodextrin and gum arabic had higher total soluble solids (10.3 and 10.4), respectively. A 54% increase in total anthocyanin content was observed for PJP with maltodextrin in comparison to waxy starch.

Keywords: Total soluble solids, oil holding capacity, particle size distribution, phenolic content, principal component analysis

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#### 1. Introduction

Pomegranate (*Punica granatum*) belongs to the Punicaceae family and is widely grown in many parts of the world, such as Europe, Asia, North Africa, the Mediterranean basin and, in the most recent time, South Africa (Fawole and Opara, 2013; Mphahlele et al., 2016). The increased commercial production of pomegranate is highly related to its rich phytochemical compounds in the edible part of the fruit (Gil et al., 2000). Polyphenols, such as flavonoids, condensed tannins and hydrolysable tannins are major components found in pomegranate arils or juice (Seeram et al., 2008). They are the major source of protective compounds against the damaging effects of free radicals (Cao, 1996). Pomegranate fruit is mostly consumed as fresh juices, flavourings and colourings and concentrates and jellies for recipes (Opara et al., 2009).

Epidemiological studies have associated the consumption of pomegranate fruit to a reduced risk of coronary heart disease, non-communicable diseases such as cancer, and diabetes as a result of its high antioxidant capacity (Malik et al., 2005; Caleb et al., 2012). Pomegranate fruit has been reported to be actively used in folk medicine, as result of its high anthocyanin compositions such as cyanidin, delphinidin and pelargonidin, which are attributed to the red colour of fruit and aril (Kulkarni and Aradhya, 2005). It is essentially useful in the cure of many parasitic diseases such as ulcers, diarrhoea, acidosis, dysentery, haemorrhage (Longtin, 2003).

Due to its health and nutritional benefits, pomegranate fruit is in demand throughout the year. Thus, the food industry desires a novel process aiming at increasing the shelf-life and improving pigment stability in pomegranate products. Drying of fruit juice in the powder form is a novel way to extend the shelf-life (Du et al., 2014). As opposed to dried pomegranate arils, pomegranate juice powders have the advantages of easier storage and distribution. Furthermore, the powders can be used as an ingredient to formulate foods. Spray drying is a commonly used method in many food industries for producing food powders and agglomerates (Sagar and Suresh Kumar, 2010; Du et al., 2014). Despite an attractive feature of this technological process, the scarce heat damage to the product is essential (Patel and Chen, 2008). Another challenging factor during spray drying is the clogging of nozzles, especially when drying sugar and acid-rich foods, such as honey and natural fruit juices (Sablani et al., 2008). The low operating conditions involved in freeze-drying could be an appropriate drying method to produce niche fruit powders from sugar and acid-rich fruit like pomegranate. Freeze-drying is a novel technique used to produce high-value powder products (Mosquera et al., 2011). It is a method that results in high-quality dehydrated products due to low operating temperatures required in the process and absence of liquid water (Jayaraman et al., 1992). This method reduces thermal damage of nutrients and preserves flavour and colour components of the product (Fazaeli et al., 2012).

Studies have reported some factors to be considered during the production of fruit powders; stickiness of powder particles, safe handling and storage (Seerangurayar et al., 2018). Stickiness during drying is mainly due to high content of sugars such as fructose, glucose, sucrose and acid materials; for example, organic acids such as citric, malic and tartaric acid which are attributed to low molecular weight and as well contribute more than 90% of solids in fruit juices (Dolinsky et al., 2000; Adhikari et al., 2004). In order to overcome the sticky behaviour of fruit juice powder, high molecular weight carriers or drying aids such as

maltodextrin, gum arabic, and waxy starch, pectin, vegetable fibres, and starches as encapsulation agents are added (Adhikari et al., 2004; Sablani et al., 2008; Osorio et al., 2011; Wang and Zhou, 2012). For instance, mango juice powder obtained through maltodextrin, gum arabic, and waxy starch resulted in characteristic amorphous particles (Cano-Chauca et al., 2005; Silva et al., 2006).

Yousefi et al. (2011), reported that gum arabic showed a high colour change and increased transition glass temperature ( $T_g$ ) of pomegranate powder. Similarly, Seerangurayar et al. (2018), reported that carrier agent-added date powders had lower hygroscopicity, which offers good storage stability. Fazaeli et al. (2012) reported that additives enhanced the properties of the final product as a result of an increase in glass transition temperature ( $T_g$ ) and high stability of quality attributes of black mulberry juice powder during storage. However, there is a limited scientific study specifically on the processing of pomegranate juice with the use of a freeze-dryer. To further examine the field of application for pomegranate products, this work investigated freeze-drying of pomegranate juice and to evaluate the influence of different carrier agents (maltodextrin, gum arabic and waxy starch) on the physicochemical, antioxidant activities and rheological properties of the powders.

## **2. Materials and methods**

### **2.1. Raw material and sample preparation**

Pomegranate fruit (cv. Wonderful) were harvested at commercial maturity from Blydeverwacht orchard, Wellington, South Africa (33°01'00" S, 18°58'59" E). Fruit were sorted for uniformity in size, shape, and colour and transported in an air-conditioned vehicle to the Postharvest Technology Laboratory at Stellenbosch University. Fruits were washed and the juice was extracted using a hand-operated domestic press.

The fresh juice was thawed and clarified using a centrifuge system (5810 R Eppendorf AG, Hamburg, Germany) at 10 000 rpm for 20 min. The cold, sterile single strength clarified juice with 16.2 °Brix (total soluble solids) was diluted and standardized with distilled water to 12 °Brix and rapidly frozen at -80 °C until experiments were carried out.

In order to obtain a flowable powder from pomegranate juice, a preliminary study was conducted to investigate the amount of carrier that would be added to the pomegranate juice. This was examined at a range between 10 to 40 g of maltodextrin (Sigma Aldrich Co., USA), gum arabic (Sigma Aldrich Co., France) and waxy starch (Sigma Aldrich Co., USA) in 100 mL pomegranate juice was carried out to select a suitable concentration of carrier agent. A 30 g concentrations of (maltodextrin, gum arabic or waxy starch) / 100 mL was observed to

produce a flowable powder, which was added after standardization. The mixture was homogenized using a laboratory homogenizer for 5 min (Yousefi et al., 2011).

## 2.2. Freeze-drying procedure

The pomegranate juice was placed in a 90 mL specimen jar and frozen in a static air freezer at -80°C. Freeze-drying of frozen samples was carried out in triplicates. Specimen jar containing samples were carefully taken to a laboratory-scale freeze-dryer (VirTis Co., Gardiner, NY, USA) operating at condenser temperature -85 °C, pressure 2.4 Pa and drying continued for 72 h. Dried samples were removed from the freeze-dryer and grounded by electrical blender to free-flowing powder. The pomegranate juice powders (PJP) were transferred and sealed in plastic bags in a desiccator containing phosphorus pentoxide to avoid moisture gain from the surrounding air until further analysis.

### 2.3. Yield, water activity and physicochemical attributes of PJP

#### 2.3.1. Powder yield determination

The percentage yield of powder was calculated based on the fresh weight (Mujaffar et al., 2015)

$$\text{Yield}(\%) = \frac{\text{Weight of powder (g)}}{\text{Fresh weight (g)}} \times 100 \quad (1)$$

#### 2.3.2. Determination of water activity and moisture content

The water activity ( $a_w$ ) of PJP was determined with an electronic dew point water activity meter (CH 8853 Novasina AG, Lachen, Switzerland). The final moisture content of the PJP was measured using a moisture analyzer (KERN DBS 60-3 Balingen, Germany) at 120°C.

#### 2.3.3. Colour measurement

Colour of PJP was determined by direct reading using a chromo-meter (Minolta model CR-200, Osaka, Japan) to obtain the colour values:  $L^*$  (brightness/darkness),  $a^*$  (redness/greenness), and  $b^*$  (yellowness/blueness). The measurements were taken at three different times from a colourless petri dish and averaged. The maximum for ' $L^*$ ' value is 100 (white), and the minimum is zero (black). The colour attributes chroma  $C^*$  and hue angle  $h^\circ$  were calculated (Fawole and Opara 2013a; Pathare et al., 2013).

$$h^\circ = \tan^{-1} \frac{b^*}{a^*} \quad (2)$$

$$C^* = \sqrt{a^{*2} + b^{*2}} \quad (3)$$

L\*, a\* and b\* represents the value after drying at each treatment levels and results were expressed as means  $\pm$  S.E. of determinations obtained.

#### **2.3.4. Determination of total soluble solids, titratable acidity and pH**

Five grams of PJP were extracted in 50 mL distilled water. The mixture was vortexed for 5 min with the use of a vortex and sonicated for 15 min in an ultrasonic bath. This was followed by centrifugation at 10 000 rpm for 25 min and recovery of the supernatant for TSS, TA and pH measurements. TSS was measured using a digital hand refractometer (model PT-32; ATAGO, Tokyo, Japan) blanked with distilled water. For TA, two millilitres of supernatant was diluted in 70 mL of distilled water and titrated against 0.2 N of sodium hydroxide (NaOH) to a pH of 8.2 using a Metrohm 862 Compact titrosampler (Herisau, Switzerland).

### **2.4. Rheological characterization of PJP**

#### **2.4.1. Solubility**

Solubility (%) was determined using the Eastman and Moore method (Cano Chauca et al., 2005) with some modifications. The sample (1 g) was homogenized in 50 mL of H<sub>2</sub>O and distilled in a vortex for 30 s. The solution was placed in a tube and centrifuged at 3000 rpm for 5 min at 25 °C. A 25 mL aliquot of the supernatant was transferred to pre-weighed Petri dishes. Then, it was immediately placed in an oven for drying at 105 °C for 5 h. The solubility was calculated as the difference in the initial and final weight divided by the initial weight.

#### **2.4.2. Hygroscopicity**

Hygroscopicity was determined, according to Ferrari et al. (2011) with slight modifications. The samples 2 g were placed inside a hermetic bottle that was controlled with NaCl saturated solution in a constant relative humidity chamber (MLR- 352 H Versatile Environmental Test Chamber, Kyoto, Japan) set at (68.9 % RH and 25 °C) (Largo Avila et al., 2015). To verify the condition for equilibrium between the samples and the environment, the weight of the samples was determined after 5 days. The hygroscopicity was expressed as % moisture (w.b).

#### **2.4.3. Bulk density**

PJP (20 g) was weighed in a 100 mL graduated cylinder then gently dropped 10 times on a rubber mat from a height of 15 cm. The bulk density was calculated by dividing the mass of the powder by the volume that occupied the cylinder (Goula et al. 2008).

#### 2.4.4. Water and oil holding capacity determination

The water-holding capacity (WHC) and oil holding capacity (OHC) of PJP were determined, according to Jalal et al. (2018) with some modifications. Twenty-five millilitres of distilled water or sunflower oil were added to 250 mg of dry sample, stirred and left at room temperature for 1 h. After centrifugation, the residue was weighed. The WHC was expressed as g of water held per g of sample, while the OHC was expressed as g of oil held per g of sample. The formula to calculate water holding capacity (WHC) is as follows:

$$\text{WHC/OHC (g/g)} = \frac{\text{residue fresh weight} - \text{residue dry weight}}{\text{residue dry weight}} \quad \text{WHC/} \quad (4)$$

$$\text{OHC (g/g)} = \frac{\text{residue fresh weight} - \text{residue dry weight}}{\text{residue dry weight}} \quad (4)$$

#### 2.4.5. Particle size distribution

The particle size was determined using a laser light diffraction instrument (Mastersizer S, model MAM 5005; Malvern Instruments, Malvern, UK). A small amount of PJP was dispersed in 99% isopropanol under magnetic agitation, and the distribution of particle size monitored during three successive measurements. The particle size was expressed as De Brouckere mean diameter, the mean diameter over the volume distribution, which is generally used to characterise a particle (Ferrari et al., 2012).

#### 2.4.6. Microstructure

The microstructure of PJP was examined using a scanning electron microscope (X-Max 51, Oxford Instruments, US). SEM images of powder were obtained from well-mixed powder samples. The samples were coated with very thin layer of gold under high vacuum condition to provide a reflective surface for the electron beam. The gold coating was carried out in a sputter coater (ACE200 LEICA Mikrosysteme GmbH, Vienna) under a low vacuum in the presence of inert argon gas. The gold-coated samples were subsequently viewed under the microscope.

### 2.5. Phenolic contents and antioxidant capacity

#### 2.5.1. Determination of total phenolic content (TPC)

TPC was determined by the Folin–Ciocalteu method using a methanolic extract of PJP. The supernatant (0.05 mL) was mixed with 0.45 mL of 50 % methanol in a test tube followed by the addition of 0.5 mL Folin–Ciocalteu after 2 min. The mixture was then vortexed and kept in the dark for 10 min before adding 2 % Na<sub>2</sub>CO<sub>3</sub> and further incubation for 40 min in the dark.

The absorbance of each sample was read at 520 nm in a UV-visible spectrophotometer (Thermo Scientific technologies, Madison, USA) against a blank containing 50 % methanol. Absorbance was compared with a standard curve (Gallic acid, 0 - 10 mg), and results were expressed as mg gallic acid equivalent per gram dry matter (mg GAE/g DM).

### 2.5.2. Total anthocyanin content

Total anthocyanin content (TAC) was quantified using the pH differential method (Wrolstad 1993). In triplicates, extract (1 mL) was mixed with 9 mL of pH 1.0 and pH 4.5 buffers separately. Absorbance was measured at 520 and 700 nm in pH 1.0 and 4.5 buffers and the result was expressed as cyanidin 3-glucoside using equation 5.

$$A = (A_{510} - A_{700})_{pH\ 1.0} - (A_{510} - A_{700})_{pH\ 4} \quad (5)$$

$$\text{Total monomeric anthocyanin (mg/mL)} = \frac{A \times MW \times DF}{\epsilon \times L}$$

where A=Absorbance,  $\epsilon$ =Cyd-3-glucoside molar absorbance (26,900), MW=anthocyanin molecular weight (449.2), DF=dilution factor, and L=cell path length (1 cm). Final results are expressed as equivalent per gram dry matter (mg C3gE/g DM).

### 2.5.3. DPPH radical-scavenging activity

The DPPH assay was carried out in triplicate, according to Fawole et al. (2013a). Briefly, aqueous methanolic extract of PJP (0.015 mL) was diluted with methanol (0.735 mL) in test tubes followed by the addition of methanolic DPPH solution (0.75 mL, 0.1 mM). The mixtures were incubated at room temperature for 30 min in the dark, and the absorbance was measured at 517 nm using a UV-vis spectrophotometer. Absorbance was compared with the standard curve (Trolox equivalent, 0 – 2.0 mM). The free-radical activity of PJP was expressed as Trolox equivalent (mM) equivalent per gram dry matter (mM TE/g DM).

### 2.5.4. Ferric-ion reducing antioxidant power (FRAP)

The antioxidant power of PJP was measured calorimetrically, according to Benzie and Strain (1996) and Fawole et al. (2013a). The FRAP working solution was freshly prepared in mixtures of 300 mM acetate buffer (50 mL), 10 mM 2,4,6-tripyridyl-s-triazine (TPTZ) (5 mL) and 20 mM ferric chloride (5 mL) at 37 °C. In triplicates, diluted aqueous methanolic PJP extracts (0.15 mL) were added to 2.85 mL of the FRAP working solution before incubation in the dark for 30 min. The reduction of the  $\text{Fe}^{3+}$ -TPTZ complex to a coloured  $\text{Fe}^{2+}$ -TPTZ complex at low pH by PJP extracts was monitored by measuring the absorbance at 593 nm.



Trolox (0 - 10 mM) was used for the calibration curve, and the results were expressed as Trolox (mM) equivalents per gram dry matter (mM TE/g DM).

## **2.6. Statistical analysis**

Data were processed with STATISTICA (Statistica 13.0, StatSoft Inc., Tulsa, OK, USA) and presented as means  $\pm$  standard error. All analysis was done in triplicates. Data was subjected to analysis of variance (ANOVA) and means were separated according to Fisher's LSD test at a level of significance of 95%. GraphPad Prism software 4.03 (GraphPad Software, Inc., San Diego, USA) was used for graphical presentations. Principal component analysis (PCA) was carried out using the XLSTAT software version 2012.04.1 (Addinsoft, France).

## **3. Results and discussion**

### **3.1. Quality attributes of pomegranate juice**

Table 1 shows the quality attributes of the pomegranate juice used for freeze-drying. It was observed that the pomegranate juice has strong acidity (pH value 3.10), which implied that it is less susceptible to microbial growth. The high TSS (15.3 °Brix) is typical of the pomegranate cv Wonderful, as it is considered as sweet-sour cultivar (Fawole and Opara, 2013c). Predominant sugars in pomegranate juice are glucose, fructose and sucrose (Fawole and Opara, 2013b), and are reported to contribute to powder stickiness during drying (Adhikari et al., 2004; Quek et al., 2007). The contents of antioxidants served as indicators for nutrient retention during the freeze-drying process. Pomegranate juice has a deep red colour as indicated by low L\*, a\*, C\* and h° values. Properties of colour are important quality indicator reflecting the sensory attractiveness; thus, the production of phenolic-rich coloured PJP was of high priority in this study.

### **3.2. Yield, water activity and colour attributes**

The effect of carrier agents on the yield of freeze-dried PJP is presented in (Table 2). The highest powder was obtained with maltodextrin (46.6 %) while waxy starch yielded the least (35.4 %). This disagrees with the study by Yousefi et al. (2011), who reported that gum arabic had the highest yield, in their study of the effect of carrier type and spray drying of pomegranate juice. This could be due to differences in the drying methods with the production of PJP. Also, the differences in the yield of PJP could be due to the configuration of the carrier agents. The least yield found in waxy starch was also noted by Yousefi et al. (2011) due to its crystalline nature.

The moisture content (MC) of freeze-dried PJP was significantly ( $p < 0.05$ ) different among carriers (Table 2). Gum arabic-added PJP had the highest moisture (1.8 %) after drying while waxy starch-added PJP had the least value of moisture (0.2 %). Similarly, water activity ( $a_w$ ) also had a significant ( $p < 0.05$ ) difference among the carrier agents (Table 2). The highest values of water activity were observed in the powder produced with gum arabic (0.49), followed by maltodextrin-added PJP which had (0.31), while waxy starch-added PJP had the least water activity (0.20).

In dried food materials, the moisture content is one of the factors related to drying efficiency (Ferrari et al., 2011). From this study, the powder produced with waxy starch had ( $p < 0.05$ ) low moisture and water activity which could perform better during storage stability than powders obtained with other drying agents. A decreased water activity prevents the growth of most bacteria, yeasts, and moulds, which are not capable of growing below 0.87, 0.88, and 0.65, respectively (Bourdoux et al., 2016). Further, Laroche et al. (2005) noted that water activity values ranging between 0.20 – 0.50 prevents microbial infestation in food powders. Daza et al. (2016) also reported that freeze-dried samples with values of water activity lower than 0.3 were less susceptible to microbial attack. Results from this study also supported the findings by Mosquera et al. (2012), who reported that the lower the critical water activity in freeze-dried strawberry powder, the better the stability during storage. Lower moisture prevents the agglomeration of particles which hinders caking of powder, thereby reducing the retention of active components and other powder properties, such as, flowability and dispersion of powders (da Silva et al., 2013). Lower moisture content in dried fruit is related to its low water activity (Hammami and René, 1997). The high moisture content and water activity observed in powder produced with gum arabic may be explained due to the difficulty of water to diffuse through the carrier agent, where crusts are formed around the surface particle (Goula and Adamopoulos, 2010).

### **3.3. Colour attributes**

Fresh pomegranate juice has favourable red colour due to the rich content of anthocyanin (Fawole et al., 2013a). Lightness ( $L^*$ ) of freeze-dried PJP was significantly ( $p < 0.05$ ) different among carrier agents (Table 2). Waxy starch appeared lighter (78.7) than maltodextrin and gum arabic, with lightness 69.0 and 64.6, respectively. According to Comunian et al. (2011), the increased lightness in powder obtained from waxy starch was as a result of the dilution effect which was a pure white colour of the carrier and could be responsible to the lighter colour of PJP. However, the darker colour of gum arabic could be responsible for the darker red colour of the powder. The effect of carrier type and spray drying

on the physicochemical properties of powdered and reconstituted pomegranate juice (cv. Malas) was previously assessed by Yousefi et al. (2011) and an increase in the values of  $L^*$  with the use of waxy starch and maltodextrin was observed in comparison to gum arabic.

The characteristic red colouration of pomegranate powder measured as  $a^*$  was also significantly ( $p < 0.05$ ) different among carrier agents (Table 2). Juice powder produced with maltodextrin had the highest redness (29.3), while gum arabic had the least value (16.3). Further, significant ( $p < 0.05$ ) difference was observed between carrier agents for Chroma ( $C^*$ ) of freeze-dried pomegranate powder (Table 2). PJP produced with maltodextrin had the highest  $C^*$  followed by those produced by waxy starch and gum arabic. Hue angle ( $h^\circ$ ) of PJP were significantly ( $p < 0.05$ ) different amongst carrier agents. PJP produced with maltodextrin had the least colour purity ( $h^\circ$ ; 0.6) closer to  $0^\circ$ , which suggests a higher degree of redness compared to PJP produced with gum arabic with highest hue angle (11.9). This suggests that changes observed in the red colour of PJP depend on the type of carrier agent (Kha et al., 2010; Du et al., 2014). Overall, the changes in colour attributes could be attributed to the addition of carrier and alteration in polyphenols during drying (Horuz and Maskan 2012).

### 3.4. Total soluble solids (TSS) and Titratable acidity (TA)

Table 3 shows the effect of carrier agents on the total soluble solids (TSS), titratable acidity (TA) and pH of PJP. PJP produced with maltodextrin, and gum arabic had higher TSS (10.3 and 10.4 °Brix), respectively than those produced with waxy starch (8.6 °Brix) (Table 3). Increased soluble solids composition observed for PJP produced with maltodextrin and gum arabic were approximately 17 %, higher than waxy starch. Similarly, there was a significant ( $p < 0.05$ ) difference amongst carrier agent in the titratable acidity (TA) of PJP (Table 3). PJP produced with maltodextrin had the highest TA (0.24 %) while powder produced with waxy starch had the least (0.18 %). Rahman and Lamb, (1991) stated that soluble solids, organic acids, amino acids, soluble pectin and mineral salts amongst several others are major chemical constituents found in fruit. Higher values of total soluble solids and titratable acidity observed in the powders produced with maltodextrin and gum arabic could be attributed to the crystalline nature of carrier agents. Carrier agents or additives are different in molecular weight compounds and crystalline configuration (Karatas et al., 1990; Bhandari et al., 1997; Shrestha et al., 2007; Yousefi et al., 2011).

### 3.5. Total phenolic and anthocyanin contents

Graphical representation of total phenolic content and total anthocyanin content observed are shown in (Fig. 1a and b). Results indicated that total phenolic content (TPC) of

freeze-dried PJP was significantly ( $p < 0.05$ ) different among carrier agents (Fig. 1a). PJP produced with maltodextrin had the highest TPC (341.8 mg GAE/g DM), whereas, PJP produced with gum arabic resulted in a lower total phenolic content (323.8 mg GAE/g DM), while the powder produced with waxy starch had the least TPC (313.3 mg GAE/g DM). Furthermore, total phenolic content was approximately 8.3 % more in the powder produced with maltodextrin than waxy starch.

As observed in this study, the carrier agents related differently with the production of PJP, which could be as a result of their structural complexities due to their soluble or insoluble nature. For instance, the nature of powder produced with maltodextrin and gum arabic appeared coarse with larger particle sizes which the specific surface area could be lower than powder produced with waxy starch with finer appearance. Results showed that the interactions might interfere with the extraction and determination of polyphenols in the powder samples. Du et al. (2014) noted the different interactions in three carbohydrate carriers (Maltodextrin, gum arabic and starch sodium octenyl succinate) in the production of persimmon pulp powders. The authors noted that carriers showing the least polyphenol retention had the smallest particle size with more surfaces exposed to oxygen, thus resulting in lower polyphenol retention. However, Tonon et al. (2009) reported that gum arabic showed greater potential compared to maltodextrin and tapioca starch with regard to polyphenol retention in spray-dried acai pulp powders. This disparity may be due to the different samples, structure of carriers, and drying conditions used.

Similarly, for the total anthocyanin content (TAC), there was a significant ( $p < 0.05$ ) difference amongst carrier agents (Fig. 1b). The powder produced with maltodextrin also had the highest (76.91 mg C3gE/g DM) of total anthocyanin content, whereas waxy starch had the least TAC (35.01 mg C3gE/g DM). Furthermore, an approximate 54 % higher in total anthocyanin content was observed for maltodextrin compared to waxy starch. Yousefi et al. (2011) investigated the use of maltodextrin, gum arabic, and waxy starch as carrier agents during the spray drying of pomegranate juice. The authors found that maltodextrin was more effective than gum arabic and waxy starch with regard to the preservation of anthocyanins which supports the result from this study. Similarly, Tonon et al. (2010), reported that the lowest anthocyanin retention was the powder produced with starch.

### 3.6. DPPH radical scavenging activity and FRAP activity

The radical scavenging activity (RSA) of PJP was significantly ( $p < 0.05$ ) different among carrier agents (Fig. 2a). The powder produced with maltodextrin had higher (33.19 mM TE/g DM), compared to gum arabic (28.45 mM TE/g DM) and waxy starch (26.96 mM TE/g DM). The lower value in powder produced with waxy starch could be traceable to the insoluble nature of the carrier. Similarly, the ferric reducing power (FRAP) of PJP was significantly ( $p < 0.05$ ) different amongst carrier agents (Fig. 2b). PJP produced with maltodextrin had the highest FRAP (6.97 mM TE/g DM) while powder produced with gum arabic had the least (5.09 mM TE/g DM). The higher value of the powder produced with maltodextrin could be traceable to the high soluble nature of the carrier agent. Tonon et al. (2010), reported that powders produced with maltodextrin showed higher antioxidant capacity in the spray-dried acai powder due to its high soluble nature which supported the results from this study. Tuyen et al. (2010) also noted that powder produced with maltodextrin increased the antioxidant capacity of spray-dried Gac powder. Further, maltodextrin as drying agent significantly increased the antioxidant capacity of spray-dried amla juice powder (Mishra et al., 2014).

### 3.7. Rheological properties

#### 3.7.1. Solubility

The solubility index is an important feature to characterize the wettability and dispersibility of powders in solutions. Solubility differed significantly ( $p < 0.05$ ) amongst the juice powders (Table 4). Powders produced with maltodextrin and gum arabic showed similar results with higher solubility (96.5 and 96.1 %), respectively, while waxy starch had the least solubility (35.4 %). The powder produced with maltodextrin and gum arabic were approximately 63.3 and 63.2 %, respectively, soluble than waxy starch. Higher values observed in powders produced with maltodextrin and gum arabic could be related to the crystalline nature of the powder. Cano-Chauca et al. (2005), in their study on spray drying of mango juice powder also recorded a higher value of up to 95 % for powders produced with maltodextrin and gum arabic which is similar to the values generated in this study. Similarly, the solubility values of pineapple and cashew juice powders were also higher, with average values of 81.56 and 95.1 %, respectively (Abadio et al., 2004; De Oliveira et al., 2009). The low solubility value observed in waxy starch reported in this study was also supported by Mishra and Rai (2006), who reported less solubility in the powder produced with waxy starch.

### 3.7.2. Hygroscopicity

Table 4 showed significant ( $p < 0.05$ ) differences amongst carrier agents in the hygroscopic nature of freeze-dried PJP. The powder produced with gum arabic had the highest hygroscopicity (12.6 %), followed by maltodextrin (10.2 %) while samples produced with waxy starch showed the least (4.7 %). Differences in the hygroscopic values could be due to the nature of the powders and the rate at which powders produced hold molecules of water from the surrounding air. The result from this study was similar to the study by Tonon et al. (2009), who reported that gum arabic showed the highest percentage of hygroscopicity in comparison with maltodextrin 10DE, maltodextrin 20DE and tapioca starch in acai powder. The authors noted that hygroscopicity of powder could be used to explain the mechanisms of water adsorption in powder materials as being related to the number of hydrophilic groups present in the structure of each carrier.

Further, a higher number of hydrophilic groups are present in maltodextrin and gum arabic, which relates to the easy absorption of moisture from the atmosphere (Gabas et al., 2007). The authors also explained that the dynamics of moisture adsorption by carbohydrate material is duly attributed to the links between the hydrogen present in water molecules and the hydroxyl groups available in the amorphous region of the substrate and the crystalline region. Similarly, a high hygroscopic nature of powder as a result of water absorbed from the surrounding air could also be used to explain the high moisture content of the powder (Du et al., 2014). However, the moisture-hygroscopicity relationship could not be generalized for all powder samples. For instance, a study by Ahmed et al., 2010 noted that hygroscopicity of spray-dried sweet potato was highly influenced by carrier agents and showed no direct relationship to varying moisture content.

### 3.7.3. Bulk density

Table 4 also showed the results of bulk density of freeze-dried PJP produced with a different carrier agent. Powders produced with maltodextrin exhibited the highest value of bulk density ( $0.77 \text{ g cm}^{-3}$ ), followed by gum arabic ( $0.74 \text{ g cm}^{-3}$ ), while waxy starch had the least bulk density ( $0.64 \text{ g cm}^{-3}$ ). Bulk density being the ‘mass of the solid particles in addition with moisture per total volume occupied by the particles, surface moisture and all pores, closed or open, in the surrounding atmosphere and is generally used to characterize the final product obtained by milling or drying’ (Barbosa-Cánovas and Juliano 2005; Yousefi et al., 2011). Further, Chegini and Ghobadian (2005) reported that powder with higher moisture content is usually associated with higher bulking weight because of the minute volume of water attracted

from the atmosphere and which is considerably denser than the dry solid material. This report is in line with the results obtained in this study. PJP produced with maltodextrin and gum arabic showed higher moisture content and higher bulk density. This also supported the findings by Ferrari et al. (2012), who related a higher moisture content with bulk density in powders produced with gum arabic as well as the mixture of both maltodextrin and gum arabic.

#### **3.7.4. Water and Oil holding capacity**

As shown in Table 4, there was a significant ( $p < 0.05$ ) difference amongst carrier agents in the water holding capacity (WHC) of freeze-dried PJP. Results showed that waxy starch had the highest WHC (1.84 g/g), while gum arabic had the least (0.25 g/g). Further, it was observed that waxy starch held water for up to 63.6 % and 86.4 % more than maltodextrin and gum arabic, respectively. The higher performance of waxy starch to hold water more than other carrier agents could be attributed to the particle structure of the carrier agents. An increase in the particle density of the powder is associated with the reduction in water holding capacity (Zhang et al., 2009). Hong and Zhang (2005) also reported similar results in their study on the effect of ultra-fine pulverization by wet processing on the particle structure of soybean dietary fibre.

There was a significant ( $p < 0.05$ ) difference amongst carrier agent in the oil holding capacity (OHC) of freeze-dried PJP (Table 4). Gum arabic had the highest values for oil holding capacity (1.96 ml/g), followed by maltodextrin with (1.64 ml/g), while waxy starch had the least (1.45 ml/g). This indicates that the higher the WHC of PJP, the lower the OHC for the studied carrier agents. The nature of the carrier agents could also be related to the OHC of PJP. A study by Chau et al. (2007) noted that particle size and processing technique such as the addition of additives or carriers could be traceable to the effective increase in oil holding capacity of powder.

#### **3.7.5. Particle size distribution**

Figure 3 shows the particle size distribution of freeze-dried pomegranate powders obtained using maltodextrin, gum arabic and waxy starch as carrier agents. A normal distribution curve was observed for all the carriers. The particles produced with waxy starch showed the highest volume (59.8 %) and the lowest particle diameter within the range of (8 – 40  $\mu\text{m}$ ). The powder produced with maltodextrin and gum arabic presented volumes of 59.3 and 52.5 % respectively, while their particle diameters ranged between (12 - 120 $\mu\text{m}$ ). Particles obtained from maltodextrin and gum arabic exhibited larger size ranges which are normal in



the case of powder analysis, since a higher proportion of the smaller particles occupies or fills up the spaces in between the larger ones. The formation of larger particles is not only attributed to agglomeration but also the molecular size of the carriers (Tonon et al., 2009). The process of agglomeration breaks down the powders' exposure to oxygen, and thereby anthocyanin pigments are protected. The higher retention of anthocyanin in powders formed with maltodextrin and gum arabic could be explained with the characteristic feature of powder agglomeration (Ferrari et al., 2011). The mean diameter of pomegranate powders obtained with maltodextrin and gum arabic were different to that of other fruit powders such as blackberry (13.0 – 34.2  $\mu\text{m}$ ), and raspberry (14.6 – 18.3  $\mu\text{m}$ ) (Tonon et al., 2011; Syamaladevi et al., 2012). This could be due to the low operating temperature of the freeze-dryer, which makes the initial phase of agglomeration easier as a result of irreversibly bound amongst particles during drying, resulting to larger particle sizes (Tonon et al., 2011). Also, according to Kurozawa et al. (2009), the solubility and flowability of spray-dried powder decreased with decreasing particle size. This tendency was observed in the solubility result presented in this study (Table 4). A direct particle size–solubility relationship was observed in the present study.

### 3.7.6. Microstructure

Figure 4 shows the scanning electron microscopy (SEM) micrographs of the powders produced with different carrier agents. The resulting powders had particles of different sizes, for all carrier agents. The use of maltodextrin and gum arabic resulted in a strong adherence of smaller particles to the surface of larger ones (agglomeration), which is in agreement with the results obtained for particle size distribution. The particles produced with maltodextrin and gum arabic were similar, showing predominantly angular shape, while particles prepared with waxy starch have a higher degree of uniformity with spherical shape as shown in Fig 4c. This result also supported the findings by Leonel (2007), who evaluated tapioca starch morphology and observed a rounded shape and smooth surface. Loksuwan (2007), also reported similar structure when investigating the morphology of tapioca starch during  $\beta$ -carotene encapsulation.

### 3.8. Principal component analysis

The results show the average of phenolic contents, antioxidant activity, rheological properties and colour coordinates of pomegranate of freeze-dried powder. The two principal components (F1 and F2) explain 100.0 % of the total data variance (Fig. 5). As observed, F1 explained 65.61 % of the total variance while F2 explained only 34.39 % of the total variability which showed that the disparity among freeze-dried pomegranate powder was described by the F1 (Fig. 5). The observations indicated that powder produced with maltodextrin and gum arabic



had higher positive scores along the F1 plane and could be associated with moisture content (MC), water activity ( $a_w$ ), total soluble solids (TSS), titratable acidity (TA), yield, total phenolic content (TPC), radical scavenging activity (RSA), total anthocyanin content (TAC), solubility, hygroscopicity, bulk density and oil holding capacity (OHC) (Table 5).

Moreover, the higher negative scores (Table 5) along F1 (Fig. 5) correspond to water holding capacity and lightness of the powder produced with waxy starch. Along F1 (Fig. 5), lower positive scores correspond to redness ( $a^*$ ), chroma ( $C^*$ ), hue ( $h^\circ$ ) and ferric reducing antioxidant power (FRAP) of freeze-dried powder produced with maltodextrin and gum arabic. Likewise, high positive scores (Table 5) along F2 is associated with hue ( $h^\circ$ ), moisture content (MC), water activity ( $a_w$ ) and oil holding capacity (OHC) of the freeze-dried powder produced with gum arabic (Fig. 5). Also, along the F2, high negative scores (as shown in Fig. 5 and Table 5) for maltodextrin could characterize the powder for having high redness ( $a^*$ ), chroma ( $C^*$ ), total phenolic content (TPC), radical scavenging activity (RSA) and ferric reducing antioxidant power (FRAP). However, lower positive scores along F2 were from freeze-dried powder from gum arabic (associated with total soluble solids (TSS), titratable acidity (TA), solubility and hygroscopicity). The lower negative scores (Fig. 5) along F2 (Table 5) were from maltodextrin and waxy starch (associated with lightness ( $L^*$ ), yield, total anthocyanin content (TAC), bulk density and water holding capacity (WHC)). The results demonstrated that PCA showed that powders produced with carrier agents (maltodextrin, gum arabic and waxy starch) resulted in significantly different properties.

#### 4. Conclusion

The use of three carrier agents (maltodextrin, gum arabic and waxy starch) in the production of freeze-dried PJP was investigated. The results indicated that maltodextrin was more effective in enhancing the yield as well as the physicochemical properties of the PJP such as colour, TSS and TA. Similarly, maltodextrin and gum arabic performed better as carriers agents in enhancing the solubility of freeze-dried PJP as compared to waxy starch. Maltodextrin was better in the preservation of phenolic content and antioxidant capacity of PJP. Therefore, it could be inferred that maltodextrin resulted in the best carrier agent that retained the biochemical activities and maintain the rheological properties in the production of freeze-dried pomegranate powder. This study has shown that maltodextrin is the most suitable carrier agent for the formulation or fortification of pomegranate-based food products such as baking, candies and ice-cream. This study reported the results of powder produced in a

laboratory-scale freeze-dryer. However, a scale-up can be investigated in order to produce, on an industrial scale, powders with similar characteristics. Moreover, further research is required to investigate the storability and optimisation of PJP.

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**Table 1**

Quality attributes of pomegranate juice (cv Wonderful) processed into powders

Quality attributes	Values (means $\pm$ SE)
TSS ( $^{\circ}$ Brix)	15.3 $\pm$ 0.07
TA (% citric acid)	1.35 $\pm$ 0.01
pH	3.10 $\pm$ 0.01
TPC (mg GAE/100 ml)	308.8 $\pm$ 6.36
TAC (mg C3gE/100 ml)	97.1 $\pm$ 2.84
L*	23.0 $\pm$ 2.21
a*	15.4 $\pm$ 1.37
C*	18.6 $\pm$ 1.44
h $^{\circ}$	34.5 $\pm$ 0.94

L\*, lightness; a\* redness; C\* chroma; h $^{\circ}$ , hue angle; TSS, total soluble solids; TA, titratable acidity; TPC, total phenolic content; TAC, total anthocyanin content.

**Table 2**

Yield, moisture content, water activity and colour attributes of freeze-dried PJP

Carrier	Yield %	MC %	a <sub>w</sub>	L*	a*	C*	h $^{\circ}$
Maltodextrin	46.6 $\pm$ 0.04 <sup>a</sup>	0.7 $\pm$ 0.02 <sup>b</sup>	0.31 $\pm$ 0.00 <sup>b</sup>	69.0 $\pm$ 1.42 <sup>b</sup>	29.3 $\pm$ 0.49 <sup>a</sup>	29.3 $\pm$ 0.48 <sup>a</sup>	0.6 $\pm$ 0.19 <sup>c</sup>
Gum arabic	40.6 $\pm$ 0.12 <sup>b</sup>	1.8 $\pm$ 0.02 <sup>a</sup>	0.49 $\pm$ 0.01 <sup>a</sup>	64.6 $\pm$ 0.39 <sup>c</sup>	16.3 $\pm$ 0.24 <sup>c</sup>	16.7 $\pm$ 0.23 <sup>c</sup>	11.9 $\pm$ 0.37 <sup>a</sup>
Waxy starch	35.4 $\pm$ 0.30 <sup>c</sup>	0.2 $\pm$ 0.02 <sup>c</sup>	0.05 $\pm$ 0.00 <sup>c</sup>	78.7 $\pm$ 1.20 <sup>a</sup>	18.6 $\pm$ 0.29 <sup>b</sup>	18.6 $\pm$ 0.29 <sup>b</sup>	4.0 $\pm$ 0.31 <sup>b</sup>

MC, moisture content; L\*, lightness; a\* redness; C\* chroma; h $^{\circ}$ , hue angle; a<sub>w</sub>, water activity. Data presented as means  $\pm$  SE in each row followed by different letters are significantly different (p < 0.05) according to Fisher's LSD.

**Table 3**

Physicochemical attributes of freeze-dried PJP

Carrier	TSS (°Brix)	TA (% citric acid)
Maltodextrin	10.3±0.17 <sup>a</sup>	0.24±0.04 <sup>a</sup>
Gum arabic	10.4±0.21 <sup>a</sup>	0.24±0.02 <sup>a</sup>
Waxy starch	8.6±0.20 <sup>b</sup>	0.18±0.01 <sup>b</sup>

TSS, total soluble solids; TA, titratable acidity. Data presented as means ± SE in each row followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.

**Table 4**

Rheological properties of freeze-dried PJP

Carrier	Solubility %	Hygroscopicity %	Bulk density (g cm <sup>-3</sup> )	WHC (g/g)	OHC (g/g)
Maltodextrin	96.5±0.09 <sup>a</sup>	10.2±0.04 <sup>b</sup>	0.77±0.01 <sup>a</sup>	0.67±0.03 <sup>b</sup>	1.64±0.01 <sup>b</sup>
Gum arabic	96.1±0.46 <sup>a</sup>	12.6±0.01 <sup>a</sup>	0.74±0.02 <sup>a</sup>	0.25±0.01 <sup>c</sup>	1.96±0.01 <sup>a</sup>
Waxy starch	35.4±0.09 <sup>b</sup>	4.7±0.07 <sup>c</sup>	0.64±0.02 <sup>b</sup>	1.84±0.01 <sup>a</sup>	1.45±0.01 <sup>c</sup>

Data presented as means ± SE in each row followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.

**Table 5**

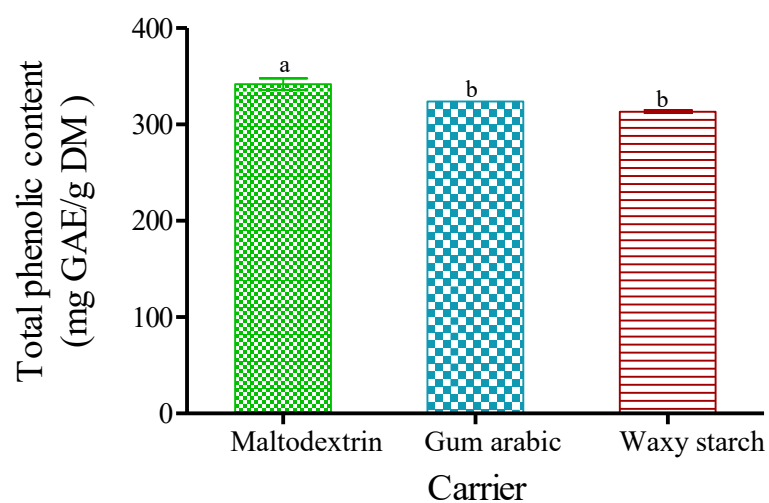
Factor loadings, eigenvalue, cumulative variance (%) and score for the first two principal (F1–F2) components based on dried pomegranate arils for hot-air and freeze-drying methods.

Loadings	F1	F2
L	-0.912	-0.411
a*	0.451	-0.893
C*	0.468	-0.884
h°	0.116	0.993
MC	0.659	0.752
a <sub>w</sub>	0.866	0.500
TSS	0.988	0.156
TA	0.994	0.107
Yield	0.898	-0.441
TPC	0.843	-0.539
DPPH	0.739	-0.674
FRAP	0.479	-0.878
TAC	0.946	-0.324
Solubility	0.995	0.103
Hygroscopicity	0.919	0.395
Bulk density	0.996	-0.088
WHC	-0.934	-0.358
OHC	0.714	0.700
Scores		
Maltodextrin	2.866	-2.842
Gum arabic	1.966	3.218
Waxy starch	-4.832	-0.376

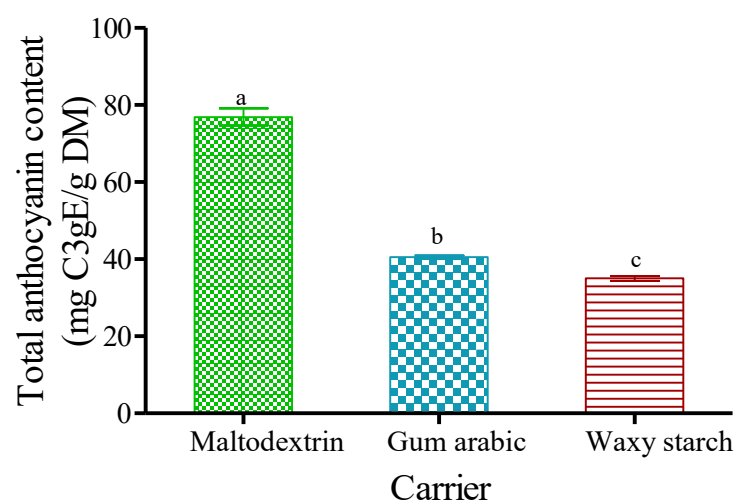
MC, moisture content; L\*, lightness; a\*, redness; C\*, chroma; h°, hue angle; RSA, radical scavenging activity; FRAP, ferric reducing antioxidant power; TPC, total phenolic content; TAC, total anthocyanin content; TSS, total soluble solids; TA, titratable acidity; a<sub>w</sub>, water activity; WHC, water holding capacity; OHC, oil holding capacity.



(a)

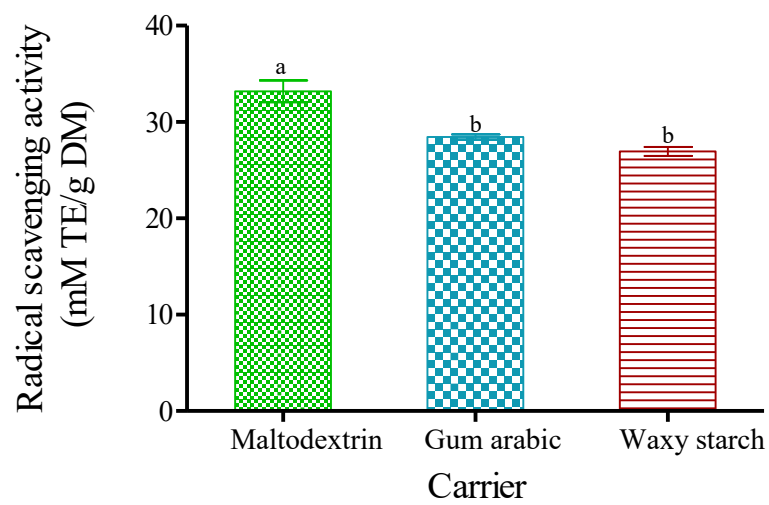


(b)

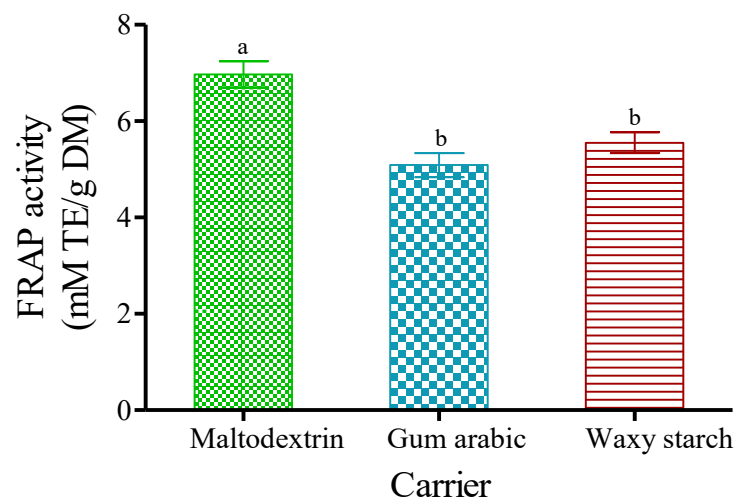


**Fig 1.** Total phenolic content (a) and total anthocyanin content (b) of freeze-dried pomegranate powder using different carriers.

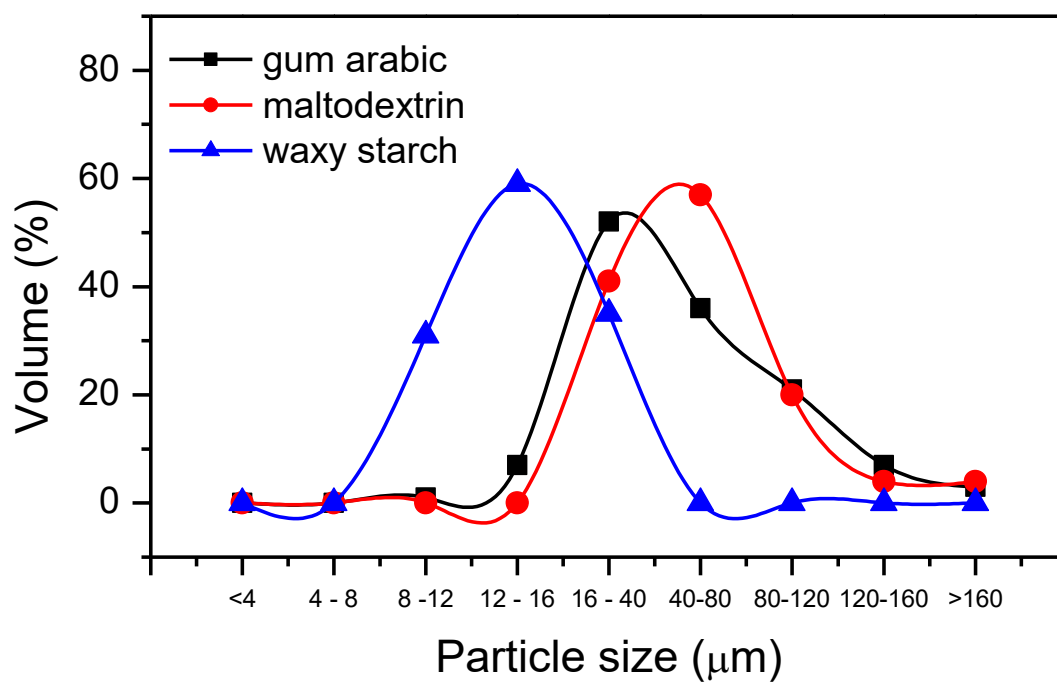
(a)



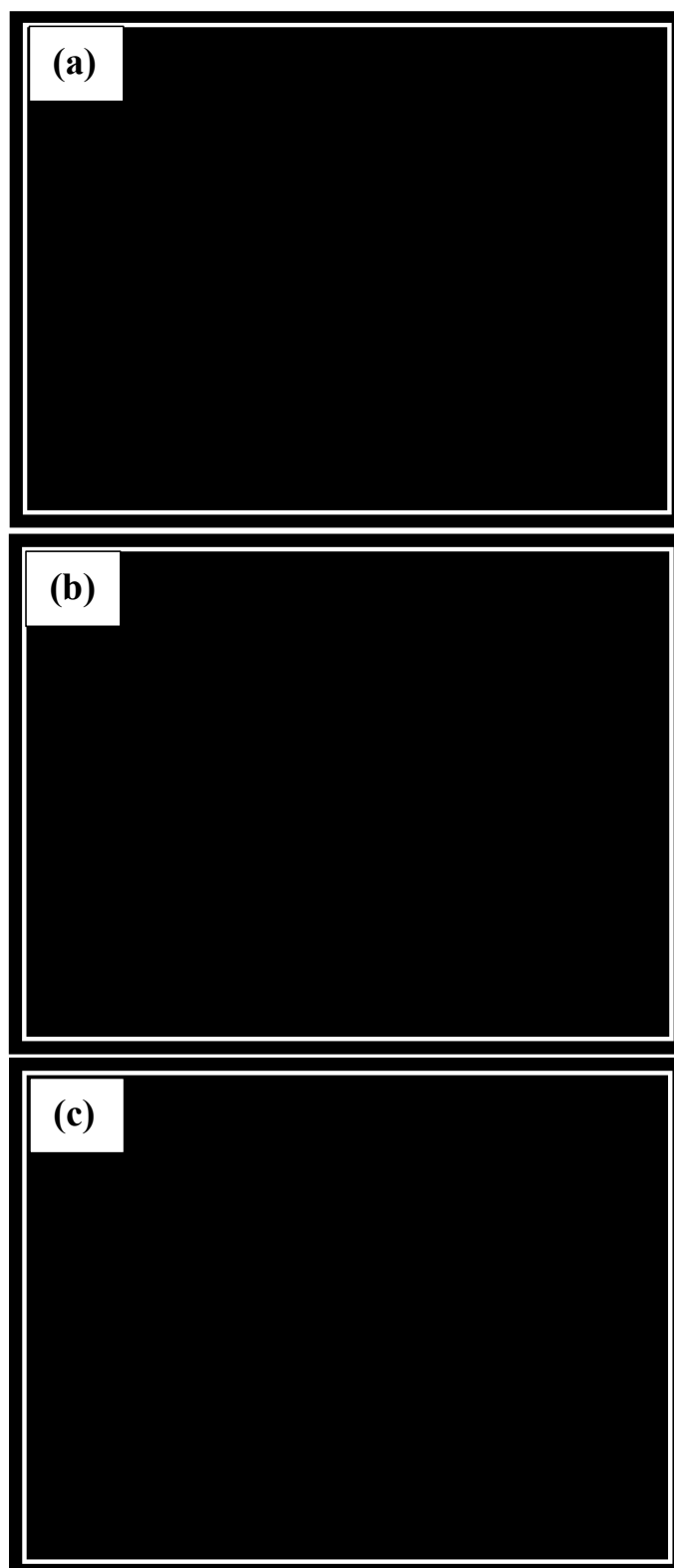
(b)



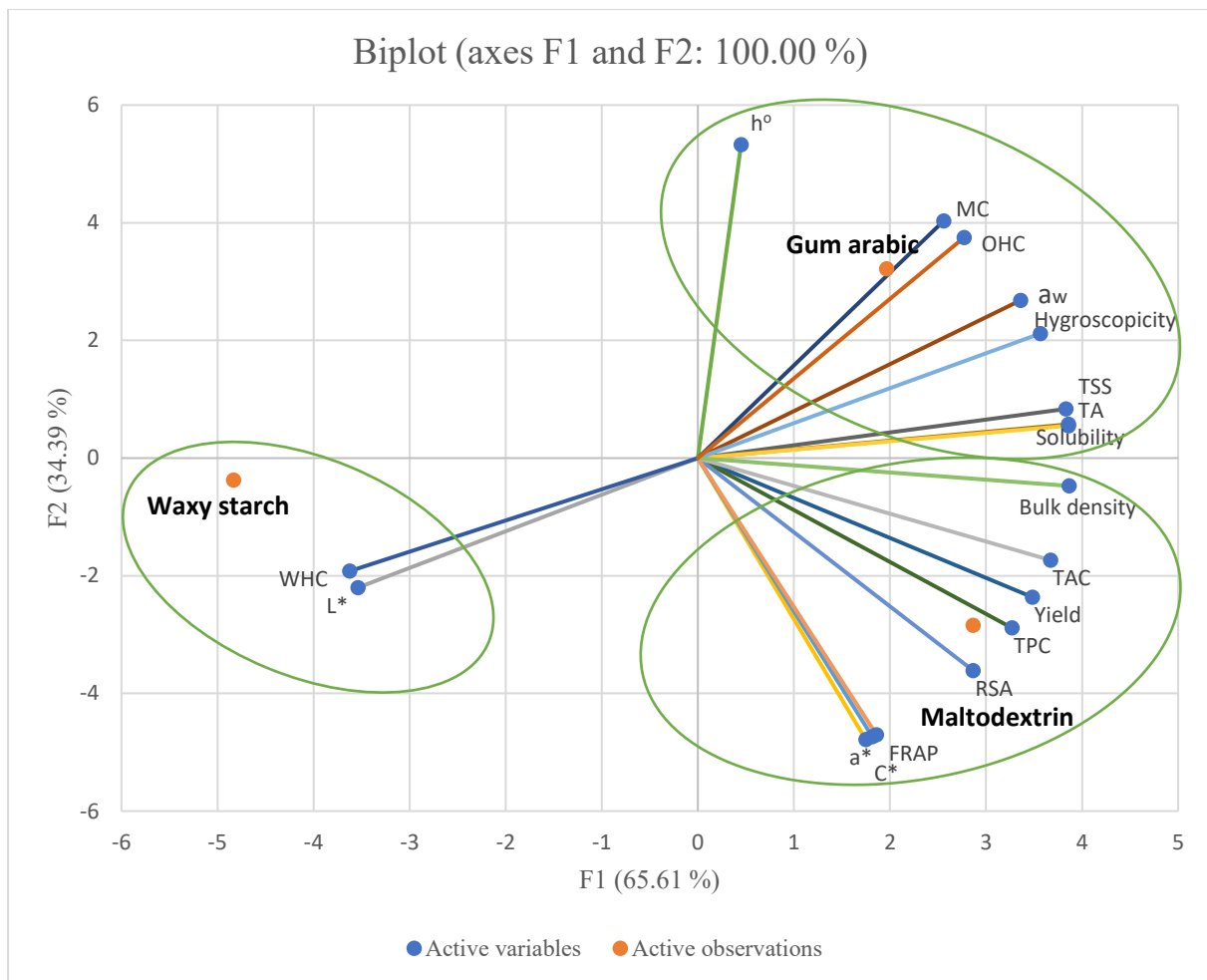
**Fig 2.** Antioxidant capacity (a) RSA and (b) FRAP activity of freeze-dried pomegranate powder using different carriers.



**Fig 3.** Particle size distribution of freeze-dried pomegranate powders produced with different carriers.



**Fig 4.** Scanning electron microscopy (SEM) microphotographs of freeze-dried pomegranate powder prepared with (a) Maltodextrin, (b) Gum arabic and (c) Waxy starch



**Fig. 5.** Principal component analysis of the first two factors (F1 and F2) based on physicochemical properties, phenolic contents, antioxidant activities and rheological properties of pomegranate powder cv. Wonderful obtained from maltodextrin, gum arabic and waxy starch. MC, moisture content;  $L^*$ , lightness;  $a^*$ , redness;  $C^*$ , chroma;  $h^{\circ}$ , hue angle; RSA, radical scavenging activity; FRAP, ferric reducing antioxidant power; TPC, total phenolic content; TAC, total anthocyanin content; TSS, total soluble solids; TA, titratable acidity;  $a_w$ , water activity; WHC, water holding capacity; OHC, oil holding capacity.

## PAPER 7

### Effect of packaging materials on quality attributes and storage-life of pomegranate (*Punica granatum*) juice powder

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#### Abstract

The effect of packaging material (aluminium foil laminated pouch (AFLP), laminated paper pouch (LPP) and polyethylene terephthalate (PET)) on the quality attributes of pomegranate juice powder (PJP) stored for 12 weeks at  $23 \pm 2$  °C and 60 % relative humidity (RH) was investigated. The physicochemical properties, total phenolic content (TPC), total anthocyanin content (TAC) and antioxidant capacity of PJP were evaluated at 4-week intervals. Moisture content and water activity of PJP increased over the storage period in all packaging materials. At the end of storage, PJP packed in AFLP had the least moisture (6.1 %), and water activity (0.22) compared to PET (7.22 %, 0.29) and LPP (7.12 %, 0.33). However, powder packed in PET had the highest total colour difference (TCD, 34.4) at the end of 12 weeks of storage, followed by LPP (30.9) and AFLP (28.0). Total phenolic content (TPC) of PJP decreased for all packaging materials after storage, from 315.3 to 217.4 mg GAE/g DM for AFLP, 315.3 to 217.4 mg GAE/g DM for LPP and 315.3 to 190.1 mg GAE/g DM for PET. The type of packaging did not significantly affect the TAC of PJP at the end of storage. However, PJP packed in AFLP had better stability of TAC as evidenced by the lowest value of rate constant ( $k$ ) and the highest value of half-life ( $t_{1/2}$ ).

Keywords: Packaging materials, total soluble solids, phenolics, antioxidants, PCA

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#### 1. Introduction

Many benefits have been attributed to fruit juice powders having high economic profitability compared to the liquid form or products, such as reduced weight, easy handling and packaging, less drudgery during transport, readily available for consumption and extended shelf-life (Phisut, 2012). The essential route to achieve this is through drying. Dried fruit products stay longer during storage, especially when dried at normal temperatures (Yousefi et al., 2011). However, most of these minimally processed foods are affected by poor postharvest handling, storage and packaging, which consequently results in shorter shelf-life (Uchekukwu-Agua et al., 2016). Several benefits are attributed to food packaging such as the ability of containment, easy communication, suitability and attraction, and the protective

measures from contamination associated with postharvest handling (Uchechukwu-Agua et al., 2015; Opara and Mditshwa, 2013).

One major postharvest challenge limiting the shelf-life and quality of juice powder is the use of inappropriate materials for packaging. Hence, the necessity to preserve fruit juice powder through the supply chain depends on optimal storage conditions and packaging materials, which will reduce quality changes in products (Zoric et al., 2017). Fruit powders and food flour samples have been reported to be suitably packed in heat-sealable laminates containing mono- or multilayers of aluminium, such as aluminium foil-laminated polyethylene (Jaya and Das, 2005), paper bags, plastic buckets and sack bags (Ogiehor and Ikenobomeh, 2006). Shishir et al. (2017) reported that processed guava powder is shelf-stable in PET pouches for ten weeks under cold or ambient conditions. An increased antioxidant property was observed in pomegranate peel powder stored with fish gelatine films (Hanani et al., 2019). According to Hymavathi and Khader (2005), the physicochemical and nutrient changes were significantly low in the mango powders packaged in metallized polyester/polyethylene compared to the polyester poly packaging.

Pomegranate (*Punica granatum*) is a fruit with high nutritional properties, which among other fruits, have enjoyed consumer's patronage within the tropical and subtropical regions of the world (Mayuoni-Kirshinbaum and Porat, 2014). It has healthy dietetic and medicinal properties as a result of a great measure of its high phenolic contents and antioxidant capacities (Fawole et al., 2012). Pigment compounds responsible for the deep red colour of pomegranate contributes about 65 % of the anthocyanin present in the fruit (Zoric et al., 2017). As a seasonal fruit, the high level of bioactive compounds in pomegranate juice, as well as the reported health benefits to date, makes it a functional ingredient in the food and nutraceutical industry (Espín et al., 2007; Fawole et al., 2012; Fawole et al., 2013).

Previous studies had mainly reported the production of pomegranate juice powder by spray drying (Robert et al., 2010; Yousefi et al., 2011; Horuz et al., 2012; Jafari et al., 2017). However, there is a need to study the storage stability of pomegranate juice powder in order to investigate the effect of different packaging materials over a long-term period. Therefore, the objectives of this study were to evaluate the effect of selected packaging materials; aluminium foil laminated pouch (AFLP), laminated paper pouch (LPP) and polyethylene terephthalate (PET) on the quality attributes and storage-life of pomegranate juice powder in ambient temperature ( $23 \pm 2$  °C, 60% RH) for 12 weeks storage period. The experimental inference

provided in this study would be beneficial to the industrial packaging and storage of freeze-dried juice powders.

## **2. Materials and methods**

### **2.1. Fruit material and processing**

Pomegranate fruit (cv. Wonderful) were harvested during commercial harvest season between February and April 2018 from an orchard in Wellington, (latitude 33°01'00" S, longitude 18°58'59" E) Western Cape Province, South Africa). Healthy fruit were sorted for uniformity in size, shape, and colour and transported in an air-conditioned vehicle to the Postharvest Technology Laboratory at Stellenbosch University. The fruit were washed and juiced using a hand-operated domestic press. The fresh juice was clarified using a laboratory centrifuge system at 10 000 rpm for 20 min. The cold, sterile single strength clarified juice was diluted and standardized with distilled water to 12 °Brix total soluble solids and rapidly frozen at -80 °C. Maltodextrin (30 %), a carrier agent was added after standardization. The mixture was homogenized for 5 min using a homogenizer Ultraturax (IKA Labortechnik, Staufen, Germany).

Pomegranate juice sample was frozen at -80 °C for 24 h in a freezer. All samples were dehydrated using a freeze-dryer (VirTis Co., Gardiner, NY, USA) at a vacuum pressure of 6 Pa and a condenser temperature of -88.7 °C for 72 h. Dried samples were removed from the freeze-dryer and grounded by electrical blender to free-flowing powder. Pomegranate powder 50 g were packed in pouches of the three different commercially available laminates: (i) AFLP, (ii) LPP, and (iii) PET (Sigma Aldrich, Johannesburg, South Africa). All samples were stored at ambient temperature  $23 \pm 2$  °C, 60% RH for 12 weeks and analyses were conducted at weeks 0, 4, 8, and 12.

### **2.2. Extraction of samples**

Extraction was carried out using 5 g of pomegranate powder in 50 mL distilled water. The mixture was vortexed for 5 min with the use of a vortex and sonicated for 15 min in an ultrasonic bath. This was followed by centrifugation at 10 000 rpm for 25 min and recovery of the supernatant for TSS, TA and pH measurements. For phenolic content and antioxidant capacity, the same procedure was followed using 50 % methanol.

### **2.3. Determination of total soluble solids (TSS), titratable acidity (TA) and pH**

TSS was measured using a digital hand refractometer (model PT-32; ATAGO, Tokyo, Japan) with the range of 0-32 °Brix, which was blanked with distilled water. For TA,



pomegranate juice supernatant (2 mL) was diluted in 70 mL of distilled water and titrated against 0.2 N of sodium hydroxide (NaOH) to a pH of 8.2 using a Metrohm 862 Compact titrosampler (Herisau, Switzerland). The pH value of pomegranate juice was measured using a pH meter (Crison, Barcelona, Spain).

## 2.4. Colour measurement

Pomegranate juice powder colour was determined by direct reading using a chromometer (Minolta model CR-200, Osaka, Japan) to obtain the colour values:  $L^*$  (brightness/darkness),  $a^*$  (redness/greenness), and  $b^*$  (yellowness). The measurements were taken at three different points from the powders placed on a transparent petri dish and averaged. The maximum for ' $L^*$ ' value is 100 (white), and the minimum is zero (black). Positive ' $a^*$ ' value is red, negative ' $a^*$ ' is green, while positive ' $b^*$ ' value is yellow and negative ' $b^*$ ' is blue. Total colour difference (TCD) was calculated using Eq. (1) (Pathare et al., 2013; Fawole et al., 2013).

$$\text{TCD} = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (1)$$

where  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$  represents the value after drying at each treatment levels and results were expressed as means  $\pm$  standard error (S.E.) of determinations obtained.

## 2.5. Phytochemical analysis

### 2.5.1. Determination of total phenolic content (TPC)

TPC of pomegranate powder was determined by the Folin–Ciocalteu method using a methanolic extract of powder (Fawole et al., 2013). The supernatant (0.05 mL) was mixed with 0.45 mL of 50 % methanol in a test tube followed by the addition of 0.5 mL Folin–Ciocalteu after 2 min. The mixture was then vortexed and kept in the dark for 10 min before adding 2 %  $\text{Na}_2\text{CO}_3$  and further incubation for 40 min in the dark. The absorbance of each sample was read at 520 nm in a UV-visible spectrophotometer (Thermo Scientific technologies, Madison, USA) against a blank containing 50 % methanol. Absorbance was compared with a standard curve (Gallic acid, 0 - 10 mg) and results were expressed as mg gallic acid equivalent per gram pomegranate dry matter (mg GAE/g DM).

### 2.5.2. Determination of total anthocyanin content

Total anthocyanin content (TAC) was quantified using the pH differential method (Wrolstad 1993). In triplicates, extract (1 mL) was mixed with 9 mL of pH 1.0 and pH 4.5

buffers separately. Absorbance was measured at 520 and 700 nm in pH 1.0 and 4.5 buffers and the result was expressed as cyanidin 3-glucoside using equation 2.

$$A = (A_{510} - A_{700})_{pH\ 1.0} - (A_{510} - A_{700})_{pH\ 4} \quad (2)$$

$$\text{Total monomeric anthocyanin (mg/mL)} = \frac{A \times MW \times DF}{\epsilon \times L}$$

where A = Absorbance,  $\epsilon$  = Cyd-3-glucoside molar absorbance (26,900), MW = anthocyanin molecular weight (449.2), DF = dilution factor, and L=cell path length (1 cm). Final results are expressed as equivalent per gram dry matter (mg C3gE/g DM).

### 2.5.3. Stability of total anthocyanin content

Studies have shown that thermal degradation anthocyanins followed first-order reaction kinetics (Zoric et al., 2017). This form of kinetic can simply be described as in Eq. (3) where c(t) is the content at time t,  $c_0$  as the initial content (mg C3gE/g powder), t as the storage period (week) and k as the first-order degradation rate constant ( $\text{week}^{-1}$ ):

$$c(t) = c_0 \cdot \exp(-k \cdot t) \quad (3)$$

The half-life of the reaction is obtained, assuming the first-order kinetics as in equation (4):

$$t_{1/2} = -\ln 0.5/k \quad (4)$$

## 2.6. Antioxidant capacity

### 2.6.1. DPPH radical-scavenging activity

The DPPH assay was carried out in triplicate, according to Fawole et al. (2013). Briefly, under dim light, aqueous methanolic extract of pomegranate powder (0.015 mL) was diluted with methanol (0.735 mL) in test tubes followed by the addition of methanolic DPPH solution (0.75 mL, 0.1 mM). The mixtures were incubated at room temperature for 30 min in the dark, and the absorbance was measured at 517 nm using a UV-vis spectrophotometer. Absorbance was compared with the standard curve (Trolox equivalent, 0 – 2.0 mM). The free-radical activity of powder was expressed as Trolox equivalent (mM) equivalent per gram dry matter (mM TE/g DM).

### 2.6.2. Ferric-ion reducing antioxidant power (FRAP)

The antioxidant power of powder was measured calorimetrically according to Benzie and Strain (1996) and Fawole et al. (2013). The FRAP working solution was freshly prepared in mixtures of 300 mM acetate buffer (50 mL), 10 mM 2,4,6-tripyridyl-s-triazine (TPTZ)

(5 mL) and 20 mM ferric chloride (5 mL) at 37 °C. In triplicates, diluted aqueous methanolic powder extracts (0.15 mL) were added to 2.85 mL of the FRAP working solution before incubation in the dark for 30 min. The reduction of the  $\text{Fe}^{3+}$ -TPTZ complex to a coloured  $\text{Fe}^{2+}$ -TPTZ complex at low pH by powder extracts was monitored by measuring the absorbance at 593 nm. Trolox (0 - 10 mM) was used for the calibration curve, and the results were expressed as Trolox (mM) equivalents per gram dry matter (mM TE/g DM).

## 2.7. Statistical analysis

Three replicates of pomegranate juice powder were carried out per treatment on each sampling day, and experimental data were analysed using Statistica software, Version 12.0 (Statistica, Statsoft, U.S.A.). Data were analysed using a two-way factorial analysis of variance (ANOVA) (factor A = storage period; factor B = packaging material). Significant differences were established at  $p < 0.05$ , according to Fisher's LSD. Data obtained were reported as mean  $\pm$  SE for each treatment.

## 3. Results and Discussion

### 3.1. Moisture content (MC) and water activity

Moisture content (MC) showed significant ( $p < 0.0001$ ) interactions with packaging material and storage period. The percentage of moisture content in pomegranate juice powder showed a staggering trend with prolonging storage for AFLP and LPP, while in PET package, a progressive increase with storage period was observed (Fig. 1a). Significantly higher moisture content was found in powder packed in PET packages (7.22 %) followed by LPP (7.12 %) after 12 weeks of storage. However, in AFLP, a slight decrease in moisture (6.10 %) was observed at the end of storage. The increased moisture content during storage could be due to the nature and type of packaging material. Moisture content increased over the storage period, which could be due to the permeability of moisture transfer within the surrounding environment into the products (Robertson, 2009). Shishir et al. (2017) noted that the moisture content of pink guava powder increased significantly during storage. Powder packed in AFLP retained the same moisture content after 4 weeks of storage. The moisture content observed in this study was within the range of 5.54 – 7.22 %, which was below 12 % reported in the literature for pomegranate juice powder (Horuz et al., 2012). Low moisture content in powder samples is a good indication that hinders microbial growth and reduces stale food tendencies (Agrahar-Murugkar and Jha, 2011). Therefore, a possible suggestion that reduced moisture in AFLP in our study could be due to the rate of moisture permeability from the surrounding air.

Water activity, which measures the amount of free water available for biochemical reactions in food materials was observed to be significantly influenced by the interaction between packaging material and storage period ( $p < 0.0001$ ) in pomegranate juice powder. Water activity showed a steady increase with prolonged storage period for all the treatments (Fig. 1b). Significantly higher water activity was observed in powder packed in LPP (0.33) followed by PET (0.29), while the least water activity value was observed in AFLP (0.22) after 12 weeks of storage. Increased water activity during storage could be an indication that more free water was available for biochemical reactions and hence, shorter shelf-life. Several studies have reported the water activities of pomegranate powders. For instance, Vardin and Yasar (2011) noted the water activity was in the range of 0.1 - 0.18 in spray dried pomegranate juice powder. Viuda-Martos et al. (2012) also noted that water activity of pomegranate juice powder which ranged between 0.139 and 0.142. In this study, the range of water activity for the 12 weeks storage period was 0.13 – 0.33. The higher values of water activity could be due to the moisture adsorbed from the surrounding environment during prolonged storage. Roustapour et al. (2012) explained the division of sorption isotherm into three parts. These include the phenomenon when water activity is below 0.2; moisture has a chemical bond with solid materials. When water activity lies between 0.2 and 0.7 indicates that the enthalpy of the liquid phase to the vapour phase is slightly higher than the heat evaporation of pure water. Conclusively, for water activity greater than 0.7, indicates that water vapour fills up the total pores of the material and evaporation occurs with the least energy.

### **3.2. Total soluble solids, titratable acidity and pH**

The total soluble solids (TSS) is directly related to the sugar content in fruit and interprets one of the flavour qualities in several fruit (Fawole et al., 2013). There was a significant ( $p = 0.0008$ ) interaction between packaging material and storage period for TSS concentration (Table 1). The staggering trend in TSS of pomegranate juice powder was observed during storage. TSS concentration decreased by 11.5, 12.4 and 15.7 % in the powders packed in AFLP, PET and LPP, respectively, at 4 weeks of storage. However, a significant increase in TSS was observed at 8 weeks storage period for all the treatments, followed by a decline at 12 weeks of storage with powder packed in PET having the highest TSS (10.2) than AFLP; 8.30 and LPP; 9.30. The increase in TSS concentration of packaging materials at 8 weeks of storage could be as a result of concentrations of sugars due to moisture loss. At the end of storage, TSS concentration declined by approximately 31.4, 15.7, and 23.1 % in powders packed in AFLP, PET and LPP, respectively. These results indicated that the application of the

three packaging materials could slow down sugar metabolism and result in prolonged storage-life to pomegranate juice powder. The result from this study was in agreement with reports by Malundo et al. (1995), who noted that a decline in TSS at the end of storage is usually accompanied by an increase in the moisture content of tomato fruit since TSS is the major component of dry matter. However, Li et al. (2009), reported an increase in TSS with storage period in their study on the effect of nano-packing of dried Chinese jujube.

Similarly, TA concentration was significantly ( $p < 0.0001$ ) affected by the interaction between packaging material and storage period (Table 1). However, TA decreased significantly with prolonged storage in the powder packed in the three packaging materials. At the end of the storage period, TA of pomegranate juice powder decreased by approximately 44.6, 36.2 and 48.9 % for AFLP, PET and LPP, respectively. The loss of acids during storage might be due to their utilization in the inversion of sugars during storage. Bhat et al. (2014) associated the decrease in the titratable acidity during storage to the utilization in the inversion of non-reducing sugars to reducing sugars. Ashebir et al. (2009) who also reported up to 45 % decrease in TA during storage of dried tomato.

There was a significant interaction between packaging material and storage period on the TSS/TA ( $p < 0.0001$ ). TSS/TA showed an increasing trend with the highest concentration observed up to 8 weeks and a slight decrease at the end of storage for powders packed in PET and LPP, while AFLP showed a staggering trend for the 12 weeks of storage. At the end of the storage period, powder packed in LPP had the highest TSS/TA (39.3) followed by PET (33.8), while AFLP had the least value (32.8). Furthermore, changes observed in the pH of pomegranate powder during prolonged storage were significantly ( $p = 0.029$ ) affected by the interaction between packaging material and storage period (Table 1). Increase in the pH value was noticed at 4 weeks of storage for powders packed in AFLP and LPP, while in PET, pH value was maintained up to 4 weeks of storage followed by an increase at 8 weeks. A general decline at the end of storage was noticed in all the packaging materials. The decrease in pH observed could be a reflection of the type and nature of the packaging materials. Flavour characteristics of pomegranate product and products from other fruit juices are influenced by the TSS/TA ratio (Ashebir et al., 2009; Mphahlele et al., 2016). The pH of the pomegranate product characterizes its acidic taste (Zarei et al., 2011).

### **3.3. Changes in colour attributes**

Retention of food colour after processing predicts the perception of consumers and the extent of quality deterioration resulting from exposure to heat (Shin and Bhowmik, 1995). The

colour attributes of pomegranate juice powder in different packaging materials after 12 weeks of storage are presented in Table 2. Lightness ( $L^*$ ) of the pomegranate juice powder increased significantly ( $p < 0.05$ ) in all the packaging materials after the 12 weeks of storage. Powder packed in PET packages had the highest  $L^*$  (71.8) followed by LPP (68.7), while powder samples packed in AFLP showed the least  $L^*$  (66.5) at 12 weeks of storage. The general increase in  $L^*$  could be as a result of residual air remaining in the package, which could cause oxidation resulting to colour change during storage. Pua et al. (2008), on the storage stability of packaged jack fruit powder using ALP and stored at 38 °C, 90% RH suggested that residual air remaining in the package bags induced total colour change due to oxidative reaction. The authors also reported that the rate of change was due to the permeability of the packaging materials to light, oxygen and water vapour.

Similarly, redness ( $a^*$ ) of pomegranate juice powder was significantly ( $p < 0.05$ ) different in all the packaging materials for the 12 weeks period of storage (Table 2). A higher  $a^*$  was observed in powders packed in PET and LPP at the end of storage period, while a decline in  $a^*$  was observed in AFLP at 12 weeks of storage. The less redness shown in the powder packed in AFLP could be due to the high moisture and oxygen barrier properties and non-transparent behaviour to light. Redness ( $a^*$ ) of pomegranate juice powder varied and in the order of PET (37.1) > LPP (35.8) > AFLP (24.8). This suggests a higher amount of anthocyanin due to the degree of  $a^*$  preserved with the use of PET compared with other packaging materials. Robert et al. (2010) reported that the anthocyanin content in spray dried pomegranate juice powder was retained in a higher amount due to the red pigments found in the powder. Similarly, Wojdyło et al. (2016) noted a high amount of anthocyanin retention due to red pigments in dried jujube fruits. TCD with storage period was significantly ( $p < 0.05$ ) affected by packaging materials. TCD at the end of 12 weeks of storage was highest in powder packed in PET (34.4), and the least was observed in AFLP pouches (28.0). The change in colour of pomegranate juice powder could partly be due to oxidative reaction as a result of residual air remaining in the packaging materials and also the nature of the packaging materials due to light permeability (Pua et al., 2008). Kumar and Mishra (2004) also reported a total colour change in mango soy fortified yoghurt powder treated under the accelerated condition as affected by packaging materials over the storage period.

### 3.4. Total phenolic content (TPC)

There was a significant ( $p < 0.0001$ ) interaction between packaging material and storage period for total phenolic content (TPC). TPC was 315.29 mg GAE/g DM at the initial period

(0 week) and declined significantly with the advancement in storage period in the powders packed in PET and LPP, while AFLP maintained its stability at 4 weeks and declined steadily with storage period (Fig. 2). AFLP had the highest TPC (217.4 mg GAE/g DM) followed by powder stored in LPP (215.8 mg GAE/g DM) after 12 weeks of storage. However, a more significant decline of TPC was observed for powder packed in PET (190.1 mg GAE/g DM) at the end of storage. This could be due to the type and nature of the packages used over prolonged time of storage. A decline in TPC is related to enzymatic oxidation (polyphenol oxidase and peroxidase) during storage (Fawole and Opara, 2013). Also, the gradual loss of phenolic content of dried food materials is associated with several factors such as, storage temperature, light and/or oxygen exposition, pH and porosity (Sablani, 2006; Sacchetti et al., 2008). Considering the nature of the PET packages, it is a type of material with complete exposure to light and could be a major factor resulting in drastic reduction in the total phenolic content during storage. Differences in TPC was also reported by Henriquez et al. (2013), on the storage stability test of apple peel using HDPE and MFHB packaging bags.

### **3.5.Total anthocyanin content (TAC)**

There was a significant ( $p < 0.0001$ ) interaction between packaging material and storage period for TAC. TAC of pomegranate powder showed a steady decrease in all treatments during storage (Fig. 3). TAC of pomegranate powder packed in AFLP, PET and LPP was not statistically different at the end of storage. However, for the TAC of juice powder packed in AFLP decreased by 88.2 %, whereas each of PET and LPP decreased by 87.8 % at the end of storage, thus, suggesting continuous anthocyanin degradation with storage period. A similar observation was reported by Fang et al. (2011) for spray dried bayberry. The authors reported up to 94 % degradation in anthocyanin during storage. Ferrari et al. (2013) also indicated that the anthocyanins in spray dried blackberry powder were more susceptible to degradation. Furthermore, differences in total anthocyanin content using different packaging materials in quamachil aril powder during storage was also reported by Rao et al. (2011). A similar observation was also noticed in litchi pericarp (Zhang et al., 2001) which could also be due to light or oxygen exposition, as well as the pore spaces in packaging bags (Sablani, 2006). Bhatia et al. (2013) also noted a decline in the anthocyanins of minimally processed Mridula pomegranate arils during storage due to the influence of packaging material.

### **3.6. Stability of total anthocyanin content (TAC) during storage**

The kinetic parameters for the changes observed in the total anthocyanin content during storage could be a valuable indicator to predict the stability of pomegranate powder and were



regarded as a function of temperature ( $23 \pm 2$  °C) and time (12 weeks storage) and calculated for isothermal experiments. The content of total anthocyanin started at week zero and was taken as a reference value to evaluate the anthocyanin stability of pomegranate powder during storage (Fig. 3). The more progressive loss was observed during storage and marked by the kinetic parameters; rate constant ( $k$ ) and the half-life ( $t_{1/2}$ ) (Table 3a). Pomegranate powder stored in different packaging materials showed the first-order reaction kinetics for TAC. Thermally degraded TAC was observed in all the packaging materials. A rapid degradation was noticed at 4 weeks of storage for the three packaging materials. For instance, as shown in Table 3b, TAC degraded up to 51.8 % after 4 weeks of storage in AFLP compared to PET (68.5 %) and LPP (60.9 %). At the end of 12 week storage period, a continuous degradation was observed up to 88.2, 87.8 and 87.8 % for AFLP, PET and LPP, respectively, (Table 3b) which reflected the non-statistical ( $p < 0.05$ ) difference shown among the packaging materials (Fig. 3). This indicated high sensitivity of TAC with prolonging storage period.

Furthermore, in the 12 weeks of storage, the lowest  $k$  value, as well as the highest  $t_{1/2}$  and  $R^2$ , was found in AFLP which indicates better TAC stability compared to PET and LPP (Table 3a, b). Zoric et al. (2017) noted that regardless of laminate type, anthocyanins are more readily degraded in sour cherry juice powder during storage. Fracassetti et al. (2013) also reported that pigments of anthocyanin are highly thermo-sensitive, and their higher degradation rate during storage increase with time and temperature. The level of degradation of pigments during processing and storage consequently change visual colour characteristics and affect the final product acceptability (Zoric et al., 2014).

### 3.7. Antioxidant capacity (RSA and FRAP)

Radical scavenging activity (RSA) was found to be significantly ( $p = 0.041$ ) influenced by the interaction between packaging material and storage period. RSA in pomegranate juice powder declined significantly with the advancement in prolonged storage for all the packaging materials (Table 4). There were approximately 17.2, 35.9 and 23.3 % decrease in the RSA for powder stored in AFLP, PET and LPP, respectively, after 4 weeks of storage. At the end of storage, about 2-, 3- and 2.5-fold decrease in RSA was observed in pomegranate powder stored in AFLP, PET and LPP packages. This is an indication that the decrease in RSA at the end of storage could be as a result of the declined total phenolic content, suggesting RSA as an attribute of phenolic compounds. A high RSA in pomegranate product is often linked to higher polyphenol concentration found in the product (Viuda-Martos et al., 2010).



Similarly, FRAP activity in the pomegranate juice powder was influenced by the combined effect of packaging material and storage period. The antioxidant (FRAP) declined significantly with the advancement in storage period for all the packaging material (Table 4). At the end of 12 weeks storage period, no significant difference was observed among the packaging materials.

A linear relationship has been reported with phenolic compounds being a major phytochemical constituent responsible for antioxidant capacity in agricultural produce (Korus, 2011). The results from this study showed that the antioxidant capacity RSA and FRAP had similar trend with total phenolic content. This result implied that the antioxidant capacity in pomegranate juice powder during storage was directly affected by phenolic compounds (Chong et al., 2013). The reductions observed in the antioxidant capacity and total phenolic content is due to the activation of oxidative enzymes such as polyphenol oxidase during the storage period (Udomkun et al., 2016). Also, the loss of total phenolic content could be closely related to factors such as storage condition, pH, as well as exposure to oxygen and light. A study by Sablani (2006) reported that high moisture content in dried material could speed up the degradation of phenolic compounds which was evident in the result of this study.

#### 4. Conclusions

Reduced moisture content in pomegranate juice powder corresponds with reduced microbial growth and hence longer shelf-stability and optimum quality attributes. Reduced water activity also resulted in a higher yield of the juice powder, which is a desirable attribute for the food industries and pomegranate juice powder processing. Pomegranate juice powder packed in AFLP provided the lowest moisture content and water activity compared to PET and LPP for the entire storage period. Based on TAC stability for the 12-week storage study, PJP was more stable in AFLP with the least rate of constant ( $k$ ) and highest half-life ( $t_{1/2}$ ) value, indicating better storage stability of PJP than PET and LPP. Overall, AFLP was more effective for long term storage of pomegranate juice powder to maintain the quality attributes essential for the food industries. Information projected from this study could be useful in packaging, storage, exporting and commercialization of pomegranate juice powder.

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**Table 1**

Effects of packaging materials and storage time on the chemical attributes of pomegranate juice powder stored at  $23 \pm 2^\circ\text{C}$ , 60% RH (dry weight basis) for 12 weeks period.

Chemical attributes	Packaging material (PM)	Week 0	Week 4	Week 8	Week 12	p values
TSS	AFLP	12.1 $\pm$ 0.09 <sup>a</sup>	10.7 $\pm$ 0.57 <sup>cd</sup>	11.0 $\pm$ 0.25 <sup>bc</sup>	8.30 $\pm$ 0.14 <sup>f</sup>	A < 0.0001
	PET	12.1 $\pm$ 0.09 <sup>a</sup>	10.6 $\pm$ 0.03 <sup>cd</sup>	11.7 $\pm$ 0.28 <sup>a</sup>	10.2 $\pm$ 0.15 <sup>d</sup>	B = 0.001
	LPP	12.1 $\pm$ 0.09 <sup>a</sup>	10.2 $\pm$ 0.20 <sup>d</sup>	11.5 $\pm$ 0.60 <sup>ab</sup>	9.30 $\pm$ 0.17 <sup>e</sup>	A x B = 0.0008
TA	AFLP	0.47 $\pm$ 0.09 <sup>a</sup>	0.24 $\pm$ 0.01 <sup>e</sup>	0.28 $\pm$ 0.00 <sup>cd</sup>	0.26 $\pm$ 0.03 <sup>de</sup>	A < 0.0001
	PET	0.47 $\pm$ 0.09 <sup>a</sup>	0.32 $\pm$ 0.00 <sup>b</sup>	0.32 $\pm$ 0.00 <sup>b</sup>	0.30 $\pm$ 0.00 <sup>bc</sup>	B < 0.0001
	LPP	0.47 $\pm$ 0.09 <sup>a</sup>	0.29 $\pm$ 0.00 <sup>bc</sup>	0.24 $\pm$ 0.00 <sup>e</sup>	0.24 $\pm$ 0.00 <sup>e</sup>	A x B < 0.0001
TSS/TA	AFLP	25.7 $\pm$ 1.22 <sup>f</sup>	44.1 $\pm$ 0.51 <sup>b</sup>	38.8 $\pm$ 0.69 <sup>c</sup>	32.8 $\pm$ 1.17 <sup>e</sup>	A < 0.0001
	PET	25.7 $\pm$ 1.22 <sup>f</sup>	33.6 $\pm$ 0.67 <sup>de</sup>	36.9 $\pm$ 1.09 <sup>cd</sup>	33.8 $\pm$ 1.00 <sup>ed</sup>	B = 0.0002
	LPP	25.7 $\pm$ 1.22 <sup>f</sup>	34.4 $\pm$ 1.12 <sup>de</sup>	48.8 $\pm$ 3.22 <sup>a</sup>	39.3 $\pm$ 1.17 <sup>c</sup>	A x B < 0.0001
pH	AFLP	3.31 $\pm$ 0.22 <sup>cd</sup>	3.53 $\pm$ 0.01 <sup>a</sup>	3.50 $\pm$ 0.01 <sup>ab</sup>	3.38 $\pm$ 0.01 <sup>bd</sup>	A < 0.0001
	PET	3.31 $\pm$ 0.22 <sup>cd</sup>	3.32 $\pm$ 0.14 <sup>cd</sup>	3.55 $\pm$ 0.01 <sup>a</sup>	3.16 $\pm$ 0.02 <sup>e</sup>	B = 0.044
	LPP	3.31 $\pm$ 0.22 <sup>cd</sup>	3.53 $\pm$ 0.03 <sup>a</sup>	3.43 $\pm$ 0.01 <sup>abc</sup>	3.24 $\pm$ 0.02 <sup>de</sup>	A x B = 0.029

Total soluble solids (TSS °Brix), titratable acidity (TA % citric acid), aluminium foil laminated pouch (AFLP), laminated paper pouch (LPP) and polyethylene terephthalate (PET). A; storage (week) and B; packaging material. Data presented as means  $\pm$  SE in each row followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.

**Table 2**

Change in colour quality of pomegranate juice powder in three packaging materials at the end of 12 weeks storage period at  $23 \pm 2^\circ\text{C}$ , 60% RH (dry weight basis).

Packaging material	$L^*$	$a^*$	TCD
Initial	$38.8 \pm 0.09^d$	$28.1 \pm 1.19^c$	-
AFLP	$66.5 \pm 0.19^c$	$24.8 \pm 0.68^d$	$28.0 \pm 0.25^c$
PET	$71.8 \pm 0.57^a$	$37.1 \pm 0.04^a$	$34.4 \pm 0.53^a$
LPP	$68.7 \pm 0.34^b$	$35.8 \pm 0.21^{ab}$	$30.9 \pm 0.37^b$

Lightness ( $L^*$ ), redness ( $a^*$ ), aluminium foil laminated pouch (AFLP), laminated paper pouch (LPP) and polyethylene terephthalate (PET). Data presented as means  $\pm$  SE in each row followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.

**Table 3a**

Effect of packaging materials on the kinetic parameters of total anthocyanin content (TAC) degradation in pomegranate powder

Packaging materials	$k \times 10^{-3}/(\text{week}^{-1})$	$t_{1/2}/\text{week}$	$R^2$
AFLP	0.1015	10.08	0.9999
PET	0.1036	9.383	0.9357
LPP	0.1112	9.357	0.9134

Aluminium foil laminated pouch (AFLP), laminated paper pouch (LPP) and polyethylene terephthalate (PET).

**Table 3b**

Degradation of total anthocyanin content (TAC) of pomegranate powder in percentage (%)

Storage (week)	AFLP	PET	LPP
	Degradation (%)		
0	-	-	-
4	51.8	68.5	60.9
8	75.9	75.0	85.9
12	88.2	87.8	87.8

**Table 4**

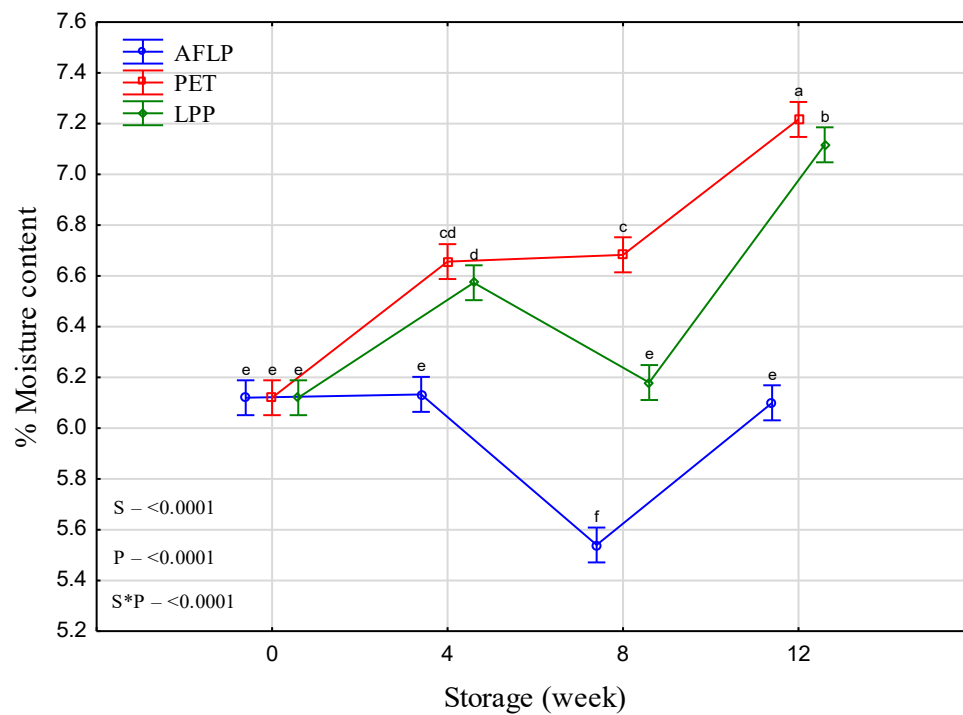
Effects of packaging and time on the antioxidant capacity (RSA and FRAP) of pomegranate juice powder for 12 weeks storage period

Chemical attributes	Packaging material	Week 0	Week 4	Week 8	Week 12	p values
RSA	AFLP	33.44±0.01 <sup>a</sup>	27.69±0.26 <sup>b</sup>	16.39±0.76 <sup>cd</sup>	18.05±2.37 <sup>cd</sup>	A = 0.0001
	PET	33.44±0.01 <sup>a</sup>	21.44±0.60 <sup>c</sup>	14.19±0.09 <sup>ef</sup>	10.77±0.60 <sup>f</sup>	B = 0.0003
	LPP	33.44±0.01 <sup>a</sup>	25.65±0.45 <sup>b</sup>	18.22±2.89 <sup>cd</sup>	13.83±1.15 <sup>ef</sup>	A x B = 0.041
FRAP	AFLP	9.84±0.01 <sup>a</sup>	6.99±0.00 <sup>c</sup>	7.67±0.02 <sup>b</sup>	6.85±0.03 <sup>c</sup>	A = 0.0001
	PET	9.84±0.01 <sup>a</sup>	6.96±0.01 <sup>c</sup>	6.69±0.00 <sup>cd</sup>	6.17±0.02 <sup>d</sup>	B = 0.028
	LPP	9.84±0.01 <sup>a</sup>	6.98±0.00 <sup>c</sup>	6.57±0.00 <sup>cd</sup>	6.54±0.00 <sup>cd</sup>	A x B = 0.138

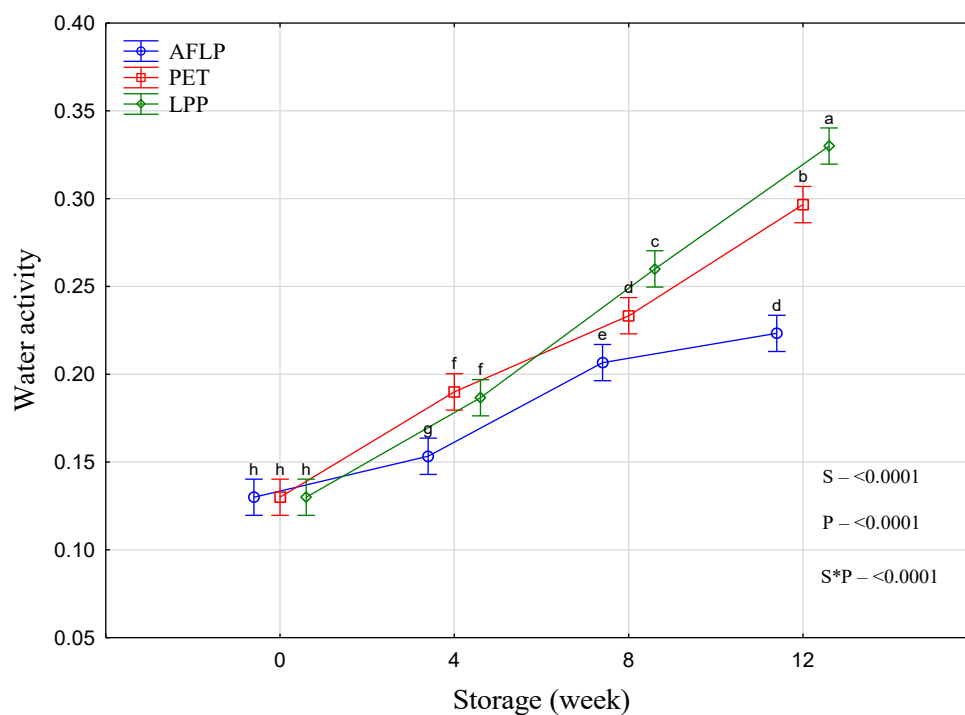
Radical scavenging activity (RSA), ferric reducing antioxidant power (FRAP), aluminium foil laminated pouch (AFLP), laminated paper pouch (LPP) and polyethylene terephthalate (PET). A; storage (week) and B; packaging material. Data presented as means ± SE in each row followed by different letters are significantly different ( $p < 0.05$ ) according to Fisher's LSD.



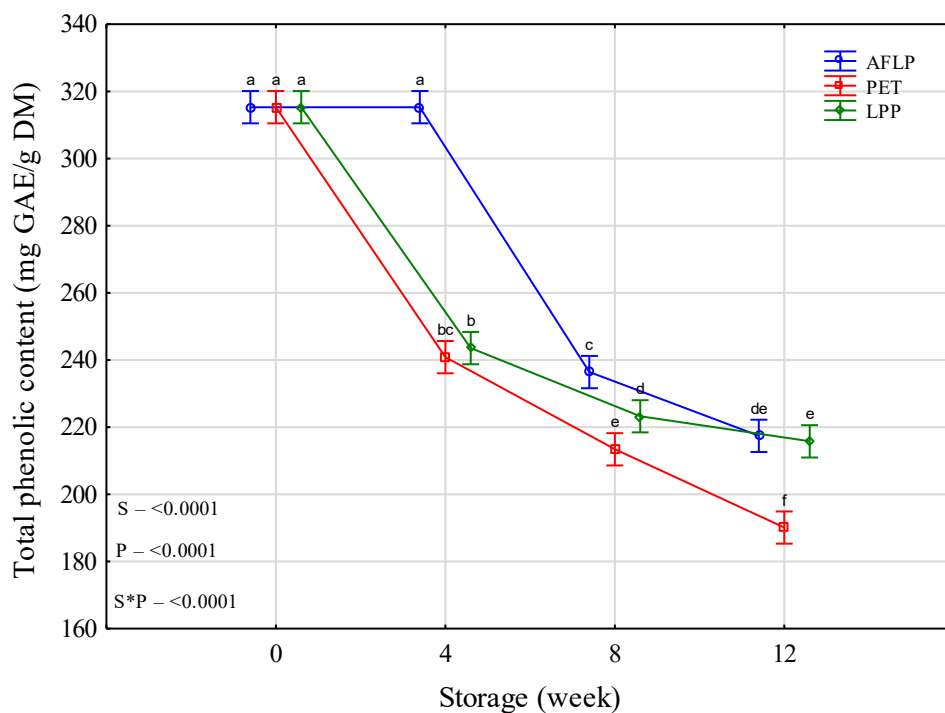
(a)



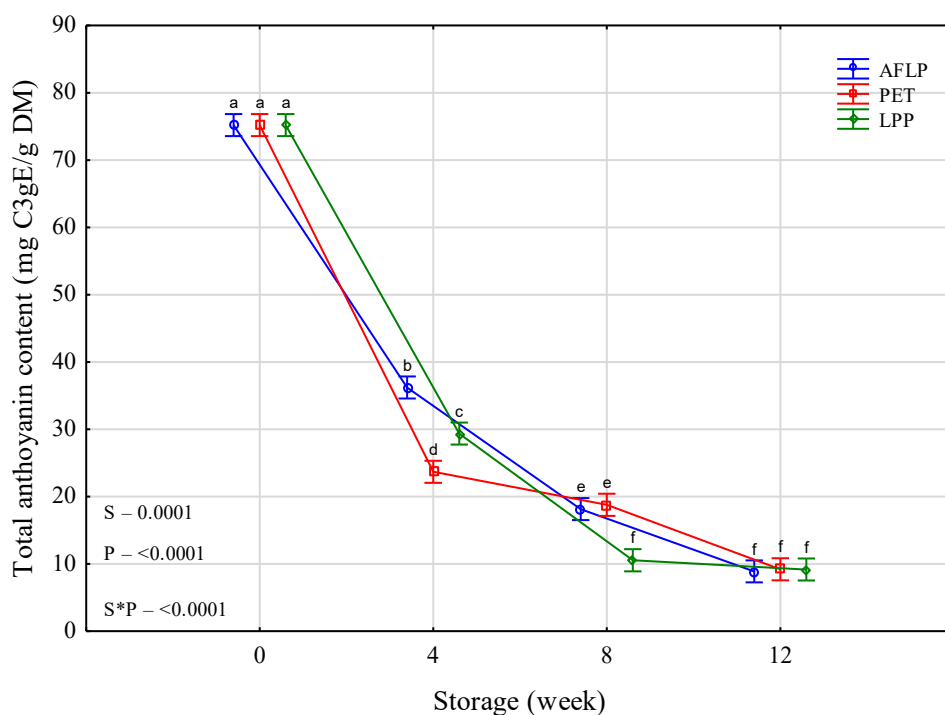
(b)



**Fig. 1.** Effects of packaging materials and storage period on the (a) moisture content and (b) water activity of pomegranate juice powder. S; storage period (week), P; packaging materials.



**Fig. 2.** Effects of packaging materials and storage time on the total phenolic content of pomegranate juice powder. S; storage period (week), P; packaging materials.



**Fig. 3.** Effects of packaging materials and storage time on the total anthocyanin content of pomegranate juice powder. S; storage period (week), P; packaging materials.

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## **GENERAL DISCUSSION AND CONCLUSIONS**

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## General Discussion and Conclusions

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### 1. Introduction

Increasing consumer awareness has led to increased consumption of fruit as fresh and processed products. This is also true for pomegranate. The health benefits of pomegranate consumption have been attributed to its exceptionally high antioxidant capacity, as a result of high concentration and unique composition of the phytochemicals in the fruit. These include flavonoids, phenolic acids and antioxidants (Opara et al., 2009; Fawole et al., 2012), but the abundant and unique, including punical acid, which is found only in pomegranate seed.

The prospects for a competitive South African pomegranate industry may be achieved not only through exportation of whole fruit but also through diversification into pomegranate agroprocessing industry in order to expand specialised production of raw material (pomegranate fruit) to support the optimisation of the health value of the processed products. It will also help to mitigate the consumption and availability of pomegranate product in the market during counter seasons. The processed products are not only a way of accommodating non-exportable grade fruit but also to supply into a high-value market for health products such as juice powder and dried arils, rich in antioxidants. This approach could offer opportunities for value addition of pomegranate fruit either by formulation or fortification into novel products, thereby positioning South Africa as a leading country in the utilising of pomegranates fruit – from fresh fruit to specialised processed products. Therefore, improved postharvest handling through different drying technologies to increase the storability and maintain the quality of a product as well as maximise potential nutritive value is of great importance. The overall aim of this research was to develop value-added and shelf-stable dried products from pomegranate aril with multiple applications and to provide science-based tools for processing and preservation of the nutritive components.

### 2. General discussion

#### 2.1. Literature review on thermal and non-thermal processing technologies of dried fruit products: pre- and postharvest implications on the quality attributes (Theme A)

This section introduced the thesis and reviewed the literature on thermal and non-thermal processing technologies of dried fruit products: pre- and postharvest implications on the quality attributes. The objective of the review was to discuss an overview of recent findings on the effects of thermal and non-thermal processing on the quality attributes of dried fruit products with a focus on cultivar, harvest maturity and storage of rawmaterial.

Fruit are known to be sources of essential nutritional diets that help to boost the immune system, detoxify contaminants, and reduce inflammation (Ames et al., 1993; Andersen and Jordheim 2006). These are greatly attributed to the phenolic compound, antioxidant activities, fibres, vitamins and mineral contents present in fruit (Andersen and Jordheim 2006; Siriamornpun et al., 2012). In addition to extending the shelf-life and maintaining the quality of fruit, the choice of drying technologies also plays a significant role (Santos and Silva 2008). The existing drying technologies can be classified as thermal and non-thermal processing methods. For instance, convective dried products at processing conditions between (27 °C – 70 °C, at air velocity 2-3 m s<sup>-1</sup>) were characterized by shortened drying time; however, instability of colour during drying was observed in hot-air dried fruit. Also, Krokida et al. (2000) reported that freeze-dryer operating at a condition ranging between -70 and -20°C and time ranging between (24 – 72 h) prevented discolouration, resulting in products with improved colour characteristics. However, before drying, several methods of pretreatment have been reported to suppress natural enzyme activity, which in turn accelerates drying and retains the quality of fruit. Some of these methods include heat (blanching and steaming) pretreatments.

Also, available evidence has shown that the proportion of bioactive compounds in fruit are influenced by pre-harvest factors including fruit variety, stage of fruit development and ripening, agronomic practices (Borochoy-Neori and Shomer, 2001; Khattab et al., 2010) and postharvest factors such as cold storage and modified atmospheric packaging of pomegranate fruit (Fawole et al., 2013; Arendse et al., 2014). However, these findings are limited to quality attributes of fruit at fresh, and minimally processed state and very few studies have reported the influence of thermal and non-thermal processing on the pre-harvest and postharvest factors of dried fruit. In addition, the need to study the effect of storage of whole fruit on quality of processed products is necessary. Therefore, this information is needed to extend the shelf-life of fruit through drying and optimise processing methods to support value addition of pomegranate products as a functional ingredient in food and nutraceuticals.

## **2.2. Characterisation of pomegranate cultivars, harvest maturity and storage of whole fruit as influenced by drying (Theme B)**

The existence of pomegranate agroprocessing industry is necessary to mitigate the challenges faced with exportation and product availability at off-season. **Papers 1** provided information on aril quality characteristics after drying. Studies on the changes that occurred in the dried arils of ‘Acco’, ‘Herskawitz’ and ‘Wonderful’ pomegranates included quality parameters such as aril colour, titratable acidity and total soluble solids. of the phytochemicals and antioxidant properties of arils after drying were also characterised. Moisture changes as

against time are usually described in terms of the transport properties of a material and the drying air (Guine et al., 2007). The behaviour of the cultivars studied during drying were different. At the end of the drying process, ‘Wonderful’ arils with the highest moisture content showed least drying times (11 h) to reach 10 % moisture, while ‘Acco’ and ‘Herskowitz’ with lower moisture content had the longest drying time (15 and 20 h), respectively, underlining in a more evident manner, the differences in the cultivar. Colour is an important attribute used for assessing acceptability, marketability and consumer preference in pomegranate (Fawole et al., 2013; Opara et al., 2009). The CIE  $a^*$  (+) value, which indicates the redness of the dried arils, ranged between 13.5 and 18.4, suggesting a colour change from light red to dark red. ‘Wonderful’ had the highest  $a^*$  value while ‘Acco’ had the least value. The observed change in aril colour parameters can be attributed to varied effects of prolonged drying, and consequently, imparting on the Maillard reactions during drying (Cinquanta et al., 2002; Sharma et al., 2013).

Optimisation amongst the investigated cultivars raised a hypothetical focus of the appropriate harvest maturity to provide the optimum marketability of the product. The scientific information on the reliable harvest maturity stage that can serve as an indicator of providing a better product from pomegranate arils is necessary. **Paper 2** provided information on the quality attribute of pomegranate arils for early (H1), mid (H2) and late harvest (H3) after drying. Also, the changes in the aril phytochemicals, and antioxidant properties after drying were also investigated. Dried pomegranate arils at mid (H2; 20.3 °Brix) and late harvest (H3; 22.2 °Brix) had significantly higher total soluble solids (TSS) than early harvest (H1; 14.8 °Brix). Also, dried arils which were harvested at mid (H2; 124.7 mg GAE/ g) had more TPC than at early (H1; 101.5 mg GAE/ g) and late (H3; 113.9 mg GAE/ g) harvest maturity stages. Zhang et al. (2009) reported a variation in the phenolic content of bitter melon leaves with maturation stages.

In practice, fruit is stored for batch processing, quality attributes degrade during the storage period, which affects the quality of processed products, it is thus important to establish the effect of long term storage of whole fruit on the quality of the final products. **Paper 3** provided information on the effect of cold storage of whole fruit on the quality of dried pomegranate arils. Freshly harvested pomegranate fruit was stored at  $7 \pm 0.3^\circ\text{C}$ ,  $92 \pm 3\%$  RH for 12 weeks and intermittent processing was carried out at 4 weeks interval. A gradual reduction in moisture was observed with storage time in whole fruit. A notable variation was observed in the total colour difference with increased storage period, with a highest total colour difference (TCD) (11.2) was observed at the end of the storage period. The significant change

in colour could be as a result of enzymatic reactions due to polyphenolic oxidase or Maillard reaction during processing (Artes et al., 1998).

### **2.3. Process optimisation of blanch-assisted dried arils and powder in relation to their quality attributes (Theme C)**

The high moisture content of pomegranate arils makes it susceptible to spoilage after harvest. However, due to a faster deterioration of arils, it was expedient to consider not only drying as a means of preservation methods but also take into consideration blanching as a method of pretreatment applied to reduce the drying time, prevent the loss of colour by inactivating enzymes and better nutritional quality retention. It is therefore important to establish the blanching conditions (temperature and time) since these factors influence the quality of finished products. Overall, results in this theme (**Papers 4 and 5**) provided valuable information to enhance better processing methods and value addition of dried pomegranate arils.

The study reported in **Paper 4** on the effect of blanching condition on the hot-air drying kinetics of three pomegranates (cvs. ‘Acco’, ‘Herskawitz’ and ‘Wonderful’) was aimed towards establishing the drying kinetics of pomegranate aril as a tool to predict drying performance. Also, a wide range of blanching (90°C for 30s, 90°C for 60s, 100°C for 30s and 100°C for 60s) as well as control (unblanched) of various cultivars were evaluated to allow for the establishment of appropriate operating conditions. Therefore, this study provides information for food processors to understand some of the phenomena taking place during drying. Seven thin layer drying models were evaluated based on the coefficient of determination ( $r^2$ ), and root mean square error (RMSE). In agreement with the findings of previous studies on mathematical modeling of dried fruit (Wang et al., 2007; Kaya et al., 2007; Minaei et al., 2012), the drying rates of the investigated blanching conditions exhibited a pattern of falling rate period, which is considered as a phenomenon of diffusion-control. However, no differences were found in the drying time, and rate for all investigated blanching conditions. For Acco cultivar, the drying behaviour was best predicted by the Logarithmic and Page model for blanched ( $R^2$  ranging between 0.9966 and 0.9989) and unblanched ( $R^2 = 0.9918$ ) samples, respectively. Furthermore, for Herskawitz cultivar, Logarithm, Page and Midili models were most suitable to predict drying behaviour for both blanched and unblanched samples. Also, for Wonderful cultivar, Logarithm and Midili models were most accurate to predict the drying behaviour for both blanched and unblanched samples amongst other models. Among the blanched conditions for the three cultivars, Logarithmic model was observed to be most common, leading to **Paper 5** with (cv. Wonderful) as a choice being the most or widely

cultivated cultivars with over 50% of total production in South Africa. **Paper 5** provided more information on the blanch-assisted hot-air drying of pomegranate arils (cv. Wonderful) with blanching conditions 90°C for 30s, 100°C for 60s and unblanched (control) samples were investigated on moisture ratio, drying rate, enzyme inactivation, physicochemical and phytochemical attributes of dried arils. Results showed that blanched samples regardless of condition reduced drying time by approximately 36.4 %, compared to unblanched samples. Unblanched arils had the least effective moisture diffusivity  $5.83 \times 10^{-9} \text{ m}^2 \text{ s}^{-1}$  compared with blanched arils at 90°C 30s and 100°C 60s with  $1.09 \times 10^{-8}$  and  $1.29 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$ , respectively. The novelty of this study involved the estimation of the residual enzyme activity in dried pomegranate arils after processing. Blanching reduced enzyme activity by 76 and 68% for 90°C 30s and 100°C 60s, respectively, compared to unblanched samples which prevent loss of colour as an important factor that affects the marketability and consumer preference of dried arils.

#### **2.4. Powder development and storability (Theme D)**

Pomegranate arils have shorter shelf-life of 5-7 days resulting in faster deterioration caused by the activity of microorganisms due to high water activity and moisture content. In a quest to develop a product with multiple applications and extended shelf-life, the conversion of pomegranate juice to powder was attempted. The research study conducted in **Paper 6** was aimed at investigating the freeze-drying of pomegranate juice and evaluate the influence of different carrier agents (maltodextrin, gum arabic and waxy starch) on the physicochemical, antioxidant activities and rheological properties of the powder. This study was conducted from a practical view of developing formulated and fortified juice powder to provide a variety of functional benefits and nutritional properties during food processing. Results obtained showed that gum arabic had the highest moisture (1.8 %) after drying while waxy starch showed the least value of moisture (0.2 %). Also, the highest values of water activity were observed in the gum arabic (0.49), followed by maltodextrin, which had (0.31), while waxy starch showed the least water activity (0.20). Freeze-dried samples with values of water activity lower than 0.3, indicated their microbial stability, as there is no microbial growth below this value (Daza et al., 2016). Maltodextrin had the highest TPC (341.8 mg GAE/g DM), while the powder formed from waxy starch had the least TPC (313.3 mg GAE/g DM).

After drying, long term storage can lead to loss of colour, nutrients and bioactive compounds. In order to maintain the quality of the dried product, packaging materials are necessary to control the amount of exposure of the product to environmental factors such as



moisture, light and oxygen. Effects of packaging materials on quality attributes and shelf-life prediction of pomegranate (*Punica granatum*) juice powder were investigated (**Paper 7**). Continuing consumer acceptance and demand for pomegranates requires that fruit be in excellent condition and exceptionally rich in nutritional and sensory quality. The aim of the study was to evaluate the effect of selected packaging materials; aluminium foil laminated pouch (AFLP), laminated paper pouch (LPP) and polyethylene terephthalate (PET) on the quality attributes and shelf-life prediction of pomegranate powder stored at ambient temperature ( $23 \pm 2$  °C, 60% RH) for 12 weeks. The study showed that significantly higher moisture content was found in individually PET package (7.22 %) followed by LPP (7.12 %) after 12 weeks of storage. However, in AFLP, a slight decrease in moisture (6.10 %) was observed at the end of storage. Furthermore, the total colour difference at the end of 12 weeks of storage was highest in PET pouches (34.4), and the least was observed with AFLP pouches (28.0). TAC degraded up to 51.8 % after 4 weeks of storage in AFLP compared to PET (68.5 %) and LPP (60.9 %).

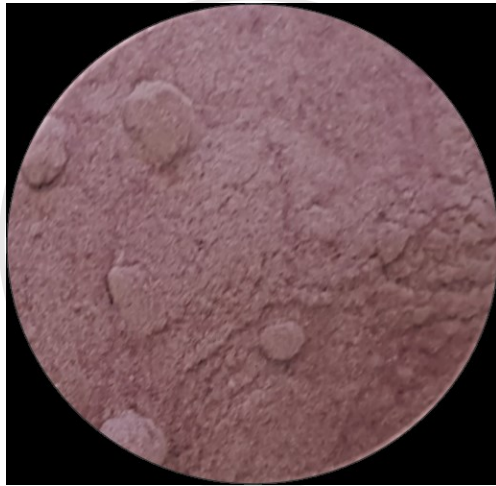
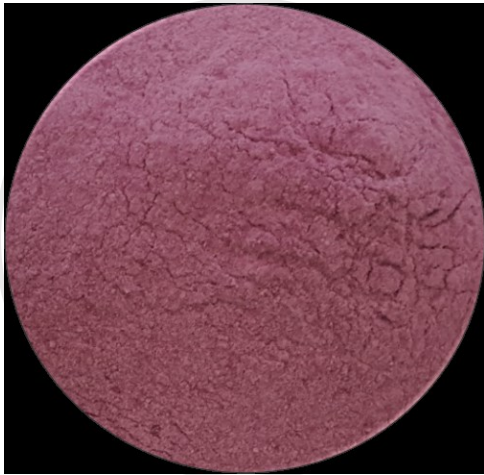
### **3. General conclusion and recommendation**

The studies reported in this thesis provides detailed information on drying of pomegranate aril and the science-based tools in optimising blanching condition and processing practices to minimise loss of nutritional properties. In this study using fruit harvested at commercial harvest maturity, ‘Wonderful’ pomegranate arils had better performance in terms of quality retention compared to ‘Acco’ and ‘Herskawitz’ after drying. However, due to the reduction in the colour and antioxidant capacity of dried arils with prolonged storage of whole pomegranate fruit, fresh pomegranate fruit are suggested for 12 weeks maximum storage time before processing. Also, based on the residual enzyme activity (PPO and POD) as well as higher anthocyanin retention, blanching at 90°C for 30s before drying at 60 °C would produce dried arils with minimal discolouration and higher phenolic content, resulting in a product with premium quality and thus recommended. Furthermore, the powder produced with maltodextrin had better quality retention compared to gum arabic and waxy starch. Pomegranate juice powder packed in AFLP provided the lowest moisture content and water activity compared to PET and LPP during the 12 weeks storage period, hence longer shelf-stability. This baseline information will assist in evaluating potential value-addition of dried products from pomegranate aril and juice for possible applications in the food and other bioprocess industries. Also, it provides a basis for future studies towards comparing blanching postharvest handling protocols to chemical and mechanical pretreatments on the commercial pomegranate cultivars grown in South Africa.

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## Appendix 1



Freeze-dried pomegranate powder produced with (a) Maltodextrin, (b) Gum arabic and (c) Waxy starch